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Field Applications of the KPEG (Potassium Polyethylene Glycolate) Process for Treating Chlorinated Wastes

PEI Associates, Inc., Cincinnati, OH

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FIELD APPLICATIONS OF THE KPEG PROCESS FOR TREATING CHLORINATED WASTES

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16. ABSTRACT

The APEG chemical dechlorination process was identified at the Franklin Research Center in Philadelphia, Pennsylvania, in 1978 for the dechlorination of polychlorinated biphenyls (PCBs) in oil. Further process development, primarily by the U.S. EPA Risk Reduction Engineering Laboratory (U.S. EPA/RREL), has focused on the dechlorination of PCBs and other potentially toxic halogenated aromatic compounds that contaminate soils. In 1987, in Moreau, New York, a pilot-scale treatment system was demonstrated on PCB-contaminated soil in batches of 35 lb each. The demonstration was the first attempt to dechlorinate PCB-contaminated soil in a reactor/mixer at a scale larger than that used in the laboratory. Analytical results of the demonstration indicated an average PCB reduction of 99.7 percent, thus illustrating the efficacy of the potassium polyethylene glycolate (KPEG) technology at a larger scale and warranting assessment for scale-up.

such as tetrachloro dibenzodioxin

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FOREWORD

Today's rapidly developing and changing technologies and industrial products and practices frequently carry with them the increased generation of materials that, if improperly dealt with, can threaten both public health and the environment. The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. These laws direct the EPA to perform research to define our environmental problems, measure the impacts, and search for solutions.

The Risk Reduction Engineering Laboratory is responsible for planning, implementing, and managing research, development, and demonstration programs to provide an authoritative, defensible engineering basis in support of the policies, programs, and regulations of the EPA with respect to drinking water, wastewater, pesticides, toxic substances, solid and hazardous wastes, and Superfund-related activities. This publication is one of the products of that research and provides a vital communication link between the researcher and the user community.

E. Timothy Oppelt, Director
Risk Reduction Engineering Laboratory

ABSTRACT

This report describes the equipment and operational procedures utilized, as well as results obtained when the potassium polyethylene glycolate (KPEG) chemical dechlorination process was implemented at several different hazardous waste sites to dechlorinate hazardous hydrocarbons. The largest KPEG treatment system described herein consisted of a steam-jacketed mixer, steam-generating plant, and condensate-collection system. On the Island of Guam, approximately 15 cubic yards of soil in batches of 1.5 to 2 cubic yards each with average initial polychlorinated biphenyl (PCB) concentrations of 3430 ppm Aroclor 1260 were treated with KPEG. PCB concentrations in the Guam soil were reduced by more than 99 percent, with no individual PCB congener exceeding 2 ppm.

In addition to the description of the field-scale KPEG process on the Island of Guam, three other cases where the KPEG process was implemented--a PCB site in Moreau, New York; PCBs in soil at the Bengart & Memel site in New York; and tetrachlorodibenzo-p-dioxin in waste herbicides at Omaha, Nebraska--are also discussed. This report also includes information regarding the preparation of the contaminated solids prior to treatment; equipment and reagents used during treatment; and analyses performed before, after, and during treatment. The report will provide the potential users of the KPEG process with sufficient information to assess which version of the KPEG process is best suited for implementation at a particular site.

This report was submitted in fulfillment of U.S. Environmental Protection Agency (EPA) Contract No. 68-03-3413, Work Assignment No. 1-2, by PEI Associates, Inc., under the sponsorship of the EPA. The project was started in June 1987 and was completed in June 1989.

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Mr. Jack Greber served as Project Director for PEI, Dr. Mike Taylor was Project Manager, and Mr. John Wentz was Assistant Project Manager. Mr. William Gallagher was lead engineer for PEI during the Moreau, New York, field evaluation of the KPEG process. Mr. Wentz was lead engineer for the design, construction, and field evaluation of the KPEG Treatment System in Guam. The diligency and dedication of PEI's field crew members (Mr. Rick Webb, Mr. David Elstun, Mr. George Byers, Mr. Todd Tullos, and Ms. Sonja Felix) is acknowledged.

The EPA's program for developing the KPEG process was led by Mr. Charles Rogers, who also was present in Guam and provided assistance and guidance during these field studies. Mr. Rogers was assisted in EPA's laboratory and field studies by Dr. Al Kornel and Mr. Harold Sparks.

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SECTION 1

INTRODUCTION

This report has been prepared for the U.S. Environmental Protection Agency (EPA) Risk Reduction Engineering Laboratory (RREL) in response to Work Assignment No. 1-2 under Contract No. 68-03-3413. The objective of this Work Assignment was to design, assemble, and field-demonstrate a field-scale chemical treatment process, also known as the KPEG process, for dechlorinating PCB-contaminated soil. This report describes the work performed by PEI Associates, Inc. (PEI) at two PCB-contaminated sites: a pilot-scale process at a site in Moreau, New York; and a field-scale process at a Navy site on the Island of Guam, U.S.A. In addition to these two field demonstrations, this report also describes two KPEG treatment demonstrations in which PCB- and TCDD-contaminated material contained in steel drums was treated in situ (in the drum in which it was stored) with KPEG reagent. The drum treatment demonstrations were performed at the Bengart & Memel site in Buffalo, New York, by Galson Research Corporation and by EPA at a site in Omaha, Nebraska.

The purpose of this report is to provide detailed information regarding equipment and procedures which have been implemented at various hazardous waste sites to degrade PCBs and another contaminant--tetrachlorodibenzo-p-dioxin (TCDD)--in contaminated solids such as soils and waste herbicides.

OVERVIEW

Halogenated chemical contaminants such as polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), and polychlorinated biphenyls (PCBs) have contaminated water, soils, and other matrices in various locations throughout the world. In laboratory animal studies, these contaminants have been found to be highly toxic; therefore, human exposure to these compounds is undesirable. To date, only limited disposal or treatment options are available for these contaminants and the matrices they contaminate--particularly soil. These large quantities of contaminated soil have created a need for a safe, cost-effective, cleanup process as an alternative to the current practices of secured on-site storage, Class B landfilling, or incineration.

In 1978, at the Franklin Research Center in Philadelphia, Pennsylvania, a chemical reagent was identified and successfully used to dechlorinate PCB-containing oils.[1] The reagent consisted of an alkali metal hydroxide (AOH)

and polyethylene glycol (PEG) mixture which became known generally as APEG (alkali polyethylene glycolate). The U.S. Environmental Protection Agency's Risk Reduction Engineering Laboratory (U.S. EPA/RREL) initiated further development of the APEG chemical dechlorination process to include soils contaminated with PCB's, PCDD's, and other potentially toxic, halogenated aromatic compounds. Initial laboratory findings indicated that PCB-contaminated soils could be decontaminated and that further investigation of the process, including assessment for full-scale remediation was warranted.

A typical laboratory-scale procedure for dechlorinating PCDD- and PCB-contaminated soil involves mixing potassium hydroxide (KOH) and polyethylene glycol-400 (PEG-400, average molecular weight of 400 Daltons) to produce the reagent known as potassium polyethylene glycolate (KPEG). The KPEG is then mixed with the contaminated soil, heated to approximately 150°C (302°F), and maintained at that temperature while continuously stirred for 1 to 4 hours. Excess reagent is then decanted, the soil neutralized with acid, rinsed two to three times with water, and the decontaminated soil discharged.

The reaction of the KOH and PEG-400 produces an alkoxide (ROK) (Equation 1), which in turn reacts with one of the chlorine atoms on the aryl ring to produce an ether ($ArCl_{n-1}OR$) and potassium chloride (KCl) salt (Equation 2). Replacement of the chlorine atom on the aryl ring with an ether-linked PEG detoxifies the compound.[2] The dechlorination process is described in Equations 1, 2a, and 2b:

$$ROH + KOH \longrightarrow ROK + H_2O \qquad (1)$$

$$ROK + ArCl_n \longrightarrow ArCl_{n-1}OR + KCl \qquad (2a)$$

$$ArCl_{n-1}OR \xrightarrow{\Delta H} ArCl_{n-1}OH + CH_2 = \underset{H}{C} - OR \qquad (2b)$$

Early APEG reagent formulations included solvents such as dimethylsulfoxide (DMSO) and triethylene glycol methyl ether (TMH). The DMSO and TMH were believed to act as cosolvents to the APEG reagent to enhance reaction rate kinetics by improving the extraction of the aryl halide wastes into the alkoxide phase.[3,4] Laboratory studies conducted by the U.S. Environmental Protection Agency Risk Reduction Engineering Laboratory (U.S. EPA/RREL) and PEI, subsequent to the first pilot-scale KPEG chemical dechlorination demonstration on PCB-contaminated soils, indicated that DMSO and TMH could be removed from the formulation without reducing the overall dechlorination effectiveness of the KPEG reagent or extending the reaction time.

PILOT-SCALE AND FIELD-SCALE KPEG TREATMENT DEMONSTRATIONS

During the summer of 1987, a pilot-scale KPEG treatment system for soils contaminated with PCB's was assembled and demonstrated by PEI at a site in

Moreau, New York. This demonstration represented one of the first attempts to implement the KPEG process for dechlorination of PCBs contaminating a soil matrix at a scale larger than that used in the laboratory. In this onsite study, four batches of PCB-contaminated soil, each weighing approximately 15.9 kg (35 lb), were treated. Initial PCB concentrations of the batches ranged from 133 to 7013 ppm and averaged 1990 ppm. PCB concentrations of the treated soil batches ranged from 1.09 to 12.4 ppm and averaged 5.6 ppm, representing an overall PCB destruction rate of more than 99.7 percent.

Subsequent to the successful application of the pilot-scale KPEG process in Moreau, New York, the U.S. EPA/RREL authorized PEI to undertake the design, assembly, and evaluation of a field-scale KPEG treatment system capable of treating PCB- and PCDD-contaminated soil in batches of 1.1 to 1.5 m^3 (1.5 to 2 cubic yards) each. The entire field-scale system was shipped to the Island of Guam, U.S.A., and was reassembled on the U.S. Navy Public Works Center (USN-PWC) site, where the field demonstration of the system was conducted. Initial PCB contaminations averaged 3430 ppm of Aroclor 1260. PCB concentrations of the treated soil were reduced by more than 99 percent, with no individual PCB congener exceeding 2 ppm.

In September 1986, Galson Research Corporation conducted a drum-treatment demonstration at the Bengart & Memel site in Buffalo, New York. The PCB-contaminated soil was treated in situ (in the 55-gallon drum in which it was stored) with KPEG reagent. The average PCB levels were reduced by 75 percent, from 108 to 27 ppm.

In addition to the drum-treatment demonstration at the Bengart & Memel site, in May 1987, the EPA conducted another drum-scale study at a site in Omaha, Nebraska. The purpose of this study was to demonstrate the effectiveness of the KPEG process for treating drummed, contaminated herbicide wastes containing TCDD. The KPEG treatment reduced the concentration of 2,3,7,8-TCDD to less than detectable concentrations, and the concentrations of both 2,4-D and 2,4,5-T were reduced by 98 percent.

The information on KPEG drum treatment demonstrations at the Bengart & Memel and Omaha sites was provided to PEI primarily in unpublished form directly from the EPA. PEI has done no laboratory testing and has not had direct access to either the EPA's laboratory or the Galson Research Laboratory. Hence, PEI can make no judgment as to the validity of these data or testing.

OBJECTIVES OF THIS REPORT

This report describes the site characteristics, procedures, and operating parameters implemented during field demonstrations of the KPEG process for treating halocarbon-contaminated soils and chemical residues. It also includes information regarding the preparation of the soil or contaminated solid residues prior to treatment; equipment and reagents used during treatment; and analyses performed before, during, and after treatment.

Detailed descriptions of four field demonstrations conducted at the Moreau, Guam, Buffalo, and Omaha sites, in which the KPEG process was implemented are presented in this report. This will assist the potential users of the KPEG process with sufficient information to assess which of the four versions of the KPEG process is best suited for implementation at a particular site. The four KPEG process demonstrations described in this report are summarized in Table 1. This table outlines the contents of the entire report in a concise manner.

TABLE 1. REPORT GUIDE

Site	Waste matrix	Contaminants treated	Reactor size	Percent reduction	Section
Moreau	Soil	PCB	40 gal	99.7	3
Guam	Soil	PCB	400 gal	99.9	4
Buffalo	Soil	PCB	Drum	75	5
Omaha	Herbicide waste	Dioxins, herbicides	Drum	98	5

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

This report will provide detailed descriptions of the equipment, reagent formulations, operating parameters, and results obtained in demonstrations of the KPEG process. KPEG treatment systems were designed and implemented to treat soils contaminated with PCBs as well as herbicide wastes containing 2,3,7,8-TCDD, 2,4-D, and 2,4,5-T. This section provides the conclusions and recommendations made on the basis of the results of each demonstration.

MOREAU, NEW YORK, PILOT-SCALE STUDY

Conclusions

The pilot-scale KPEG demonstration performed in Moreau, New York, was the first attempt to dechlorinate PCBs in a reactor/mixer at a scale larger than that used in the laboratory. Results of the demonstration indicated that in batches of 13 kg (35 lb), PCBs could be reduced by an average of 98.3 percent (93.9 to 99.8 percent).

The analytical results indicated that scale-up of the KPEG treatment system was feasible. The rocking pipe mixer, however, was determined to have limited scale-up potential.

Recommendations

From the pilot-scale KPEG demonstration, the following recommendations were made:

1. Attempt second generation scale-up to 1.15 to 1.53 m^3 (1.5 to 2.0 cubic yards) capacity.

2. Identify alternative mixer/reactor mechanism of sufficient ruggedness to perform under adverse conditions and be amenable to further scale-up.

3. Identify an alternative heat source to replace costly electrical heating.

4. Perform laboratory experiments to develop a KPEG formula which does not entail use of DMSO and TMH.

5. Provide detailed attention to materials handling since quantities were approaching the limits feasible for manual labor.

6. Assemble the complete system and perform shakedown in a familiar setting before transporting to a remote field location.

7. Design system in sections for ease of assembly, transport and disassembly.

8. Design system as labor intensive in order to learn intimate details of process and operation.

GUAM, USA, FIELD-SCALE STUDY

Conclusions

From the field-scale KPEG demonstration performed in Guam, the following conclusions have been drawn:

1. The portability and ruggedness of the equipment were proven.

2. Overall performance of the system was exceptional. Only minor difficulties and problems were encountered, as is the case with any system startup, that were readily resolvable.

3. The process was successful in its treatment of PCB-contaminated soil without the use of DMSO or TMH.

4. Most importantly, the efficacy of the KPEG technology was demonstrated when soils with initial PCB concentrations of 3535 ppm (3260 to 3828 ppm) were reduced to 6.74 ppm (1.01 to 13.9 ppm), representing a PCB reduction of 99.84 percent (99.58 to 99.98 percent), with no resolvable PCB congener exceeding 2 ppm.

5. PCBs filtered from the soil did not relocate into the condensate.

Recommendations

The following recommendations have been made concerning the field-scale KPEG system:

1. Utilize the system at additional sites to provide more background data on the technology.

2. Perform additional field-scale demonstrations to attempt to optimize the reagent formulation and operating parameters.

3. Reduce the labor intensity of the system by modifying with materials handling equipment and automation.

4. Identify alternative equipment manufacturers that may be capable of further increasing the efficiency of the system.

5. Initiate design of a full-scale, full-portable, self-supportive KPEG treatment system for soils contaminated with halogenated aromatic compounds.

6. Initiate efforts to delist the KPEG-treated soil that is "clean" per the requirements of the R&D permit.

BUFFALO, NEW YORK, DRUM-SCALE STUDY

Conclusions

The KPEG process implemented at the Bengart & Memel site was successful in reducing levels of PCBs in soil contained in 55-gallon drums to below the 50-ppm control limit set for the soil at the site. The efficacy of the process is supported by data indicating the reduction of PCBs from 108 to 27 ppm (a 75 percent reduction). PCB levels in the soil in one drum with an initial concentration of 1300 ppm were reduced by 93 percent to 78 ppm.

Recommendations

Laboratory studies and other field tests have been performed subsequent to this demonstration which have indicated that the KPEG reaction to degrade PCBs proceeds in the absence of DMSO. Since there are a number of sites with many drums that contain contaminated material, it is appropriate to perform further studies to develop a drum-scale process that utilizes a KPEG formulation prepared from only KOH and PEG. The treated soils from Bengart & Memel were approved for return to the site by the New York State Department of Environmental Quality; however, the site owner decided to dispose of the soil in a local sanitary landfill. For future applications, the regulations governing disposal of treated soils must be clarified.

OMAHA, NEBRASKA, DRUM-SCALE STUDY

Conclusions

The drum-scale KPEG process was found to be capable of degrading 2,3,7,8-TCDD, 2,4-D, and 2,4,5-T. The contaminants were contained in a 55-gallon drum and treated using only PEG and KOH. Initial concentrations of 1.3 ppm 2,3,7,8-TCDD, 17,800 ppm 2,4-D, and 2,800 ppm 2,4,5-T were reduced to concentrations of none detectable, 334 ppm, and 44 ppm, respectively. The demonstration also proved the efficacy of the KPEG process without the use of DMSO or TMH.

Recommendations

At this point in time, it is recommended that small sites with contaminants other than PCBs be included in future feasibility studies.

SECTION 3

PILOT-SCALE DEMONSTRATION OF KPEG PROCESS IN MOREAU, NEW YORK

BACKGROUND

In June 1987, PEI Associates, Inc., in cooperation with Galson Research Corporation,* under contract to EPA's Risk Reduction Engineering Laboratory, conducted a pilot-scale field demonstration in Moreau, New York, to evaluate the effectiveness of the KPEG treatment process for chemical destruction of PCBs contained in a soil matrix. In laboratory studies, the KPEG process had been found to degrade PCBs in small (less than 500-gram) aliquots of soil from the site. Since the laboratory-scale experiments yielded promising results, the data from these experiments provided a basis for scale-up, and a decision was made to use a 40-gallon reactor in this pilot-scale study.

The objectives of the demonstration at the Moreau site were 1) to document that the KPEG reagent could effectively degrade PCBs to acceptable levels at the 40-gallon scale (from 100's to 1000's of ppm to less than 3 to 4 ppm); and 2) to gather process data that would subsequently be used to design a larger (2-yd^3 or 400-gallon) reactor. The test at Moreau was not intended to be a site cleanup; only a small portion of the contaminated soil was treated as part of the test.

SITE DESCRIPTION/SITE PREPARATION/MATERIALS HANDLING

Formerly a drag strip, the Moreau site is located in a sparsely populated area of South Glenns Falls, New York, about a 1-hour drive west of Albany. The topography of the area is flat with small trees and fields. The drag strip was oiled periodically, and some of the oil used for this purpose was transformer oil containing PCBs. The PCB concentrations in the soil at the site ranged from nondetectable up to tens of thousands ppm. The site is now closed to the public and is secured by fencing and locked gates. The nearest residents live about a half-mile from the site.

An area in which PCB-contaminated soil had previously been mixed by rototilling was considered a "hot zone," and this was the area from which soil was collected for the test runs. A trailer containing the reactor and a

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* During this project, Galson performed as a subcontractor to PEI under EPA Contract No. 68-03-3413, Work Assignment 0-2.

small laboratory was placed in a clean area near the hot zone. The laboratory contained a gas chromatograph with electron-capture detector (GC/ECD), which permitted analysis for PCBs during the testing.

DESCRIPTION OF PROCESS EQUIPMENT

Figure 1 is a diagram showing details of the pipe reactor* used during the pilot-scale tests. The reactor was constructed from a 40-inch length of 16-inch steel pipe and was mounted on a stand that allowed rotation about the indicated pivot point. The reactor rotated through an arc of 180 degrees in about 10 seconds; therefore, mixing inside the pipe was gentle. A baffle along the side of the pipe (midway down its length) redirected the flow of slurry along the wall of the pipe and enhanced mixing. Another baffle, semicircular in shape and located near the flanged end of the pipe, covered about half the pipe area. The purpose of this baffle was to prevent rocks and other heavy objects from falling directly onto the flange and damaging the screen assembly which was an integral part of the flange.

The pipe had an end plate welded on one end and a 16-bolt flange on the other end. The flanged end was tapped and piped to a tee. Each bull of the tee led to a shutoff valve. One shutoff valve was capped, and the other shutoff valve went to a hose connection used for adding and draining liquids from the reactor. A screen stack consisting of a 16-mesh screen followed by a 20-mesh screen was located on the top flange. This screen stack was used to strain reagent from the soil after the reaction.

A 0° to 150°C mercury thermometer was located in the side of the pipe, just below the baffle. It was inserted through a ball valve that was used to sample the reactor contents. To take a sample, the reactor was first placed in the upright position so that the reactor contents would be at the bottom of the reactor. The thermometer was then unscrewed, and the reactor was tipped over to allow slurry to flow to the valve. After the samples were collected in a porcelain-lined pan, the valve was closed off. The reactor was then rotated back to the upright position, the valve reopened, and the thermometer reinserted.

A tap near the center of the reactor pipe was connected to a four-way connector. One of the four-way connector openings was equipped with a thermocouple. The second opening was connected to a 0- to 60-psig pressure gauge. The third opening was connected to a valve, and the valve was connected to a flexible hose that served as the vent line during the tests.

Heat was supplied to the reactor by four electric heating straps. Each heating strap was wired through a rheostat so the voltage could be controlled. A 1000-watt heating strap was wrapped around the top of the

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* The pipe reactor was assembled and operated by Galson Research Corporation. Galson holds a patent covering the use of a DMSO/KPEG (U.S. Patent No. 4,574,013, March 4, 1986) to treat PCB-contaminated soils.

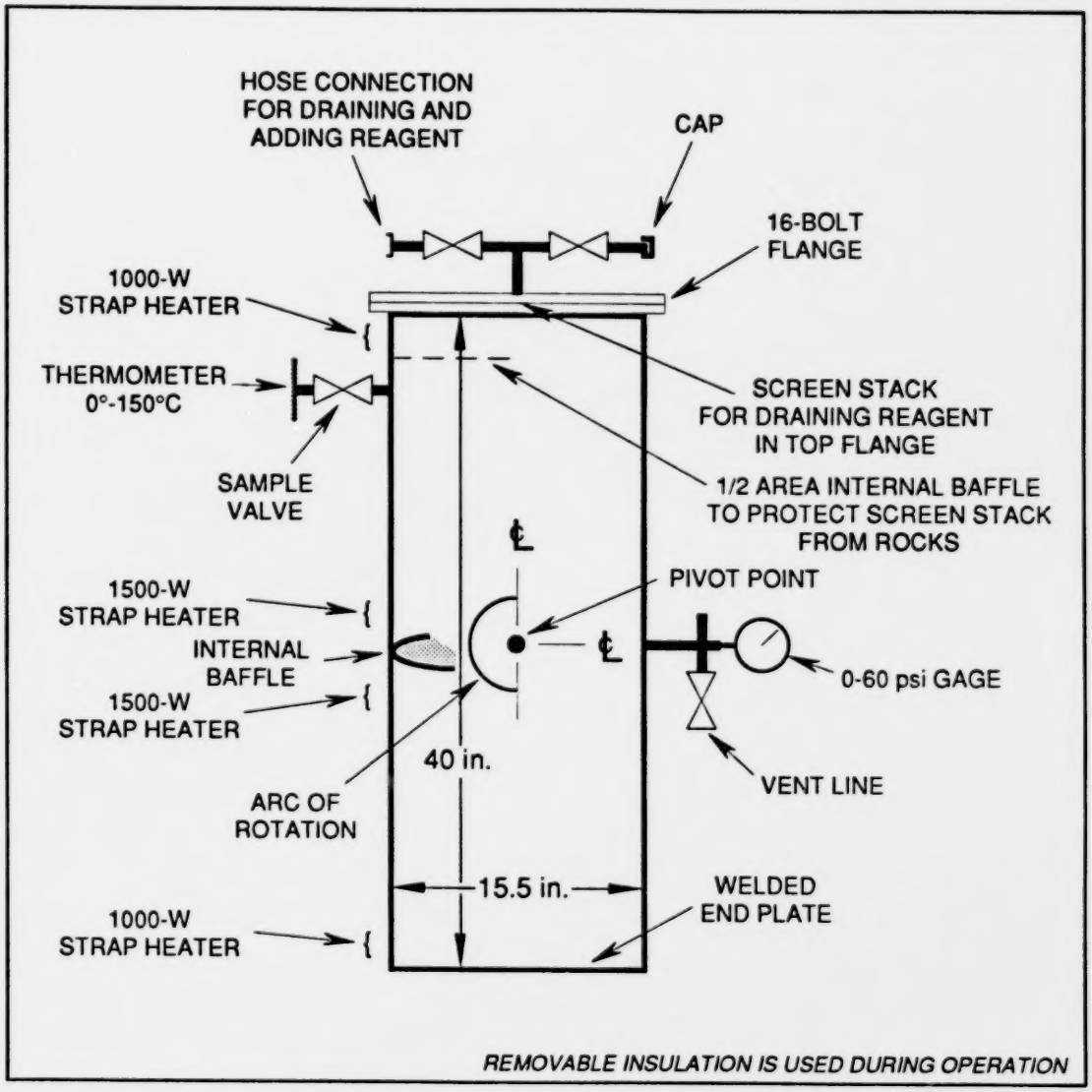


Figure 1. Reactor detail, Moreau, New York, pilot-scale demonstration.

reactor, and a second 1000-watt strap was wrapped around the bottom. Both straps were about 10 inches wide. Two 3-inch-wide 1500-watt straps were wrapped around the center portion of the reactor.

During the reactor operation, temperature and pressure were recorded every 15 minutes. In addition, every 15 minutes the reactor was stopped in the vertical position and pounded with a rubber mallet to dislodge any contents that might have stuck to its wall. The reactor motion and direction of motion were controlled by a hand-held switch; therefore, during the entire time the reactor was operating, someone had to hold the switch and manually reverse the reactor's direction at the end of each 180° turn.

Figure 2 shows the reactor vent system. Vapor from the reactor was vented through a small fan cooled condenser. The condenser condensate was collected in Condensate Drum No. 1 which would vent to an ice condenser. The ice condenser consisted of a coil submerged in an ice bath which drained to Condensate Drum No. 2. Condensate Drum No. 2 vented through an activated carbon canister for removal of any trace volatiles before atmospheric discharge.

PROCESS IMPLEMENTATION AND OBSERVATIONS

The following procedure presents the individual steps involved in treating the PCB-contaminated soil with the KPEG reactor described in the preceding subsection.

1) Place 45.4 kg (100 lb) of dimethylsulfoxide (DMSO), 22.9 kg (50.5 lb) of triethylene glycol monomethylether (TMH), and 27.7 kg (50 lb) of polyethylene glycol 400 (PEG-400) in a 55-gal drum and mix with a recirculating drum pump.

2) Load 35 lb of soil (moisture content 14 to 18%) into the reactor and add 23 lb of the solution prepared in Step 1 and 12 lb of a 45 percent KOH solution.

3) Close the reactor by bolting on the end flange after verifying that the vent line for the reactor is open.

4) Begin the rotation of the reactor and bring the temperature of the contents up to 150°C. Manually activate the switch to cause the reactor to rotate in the reverse direction every 10 seconds.

5). Withdraw samples of the reactor contents by removing the thermometer and pouring the slurry (approximately 50 g) out of the thermometer insertion port. Analyze for PCB content by using the PCB analysis method developed from EPA-600/4-81-045, "The Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils."

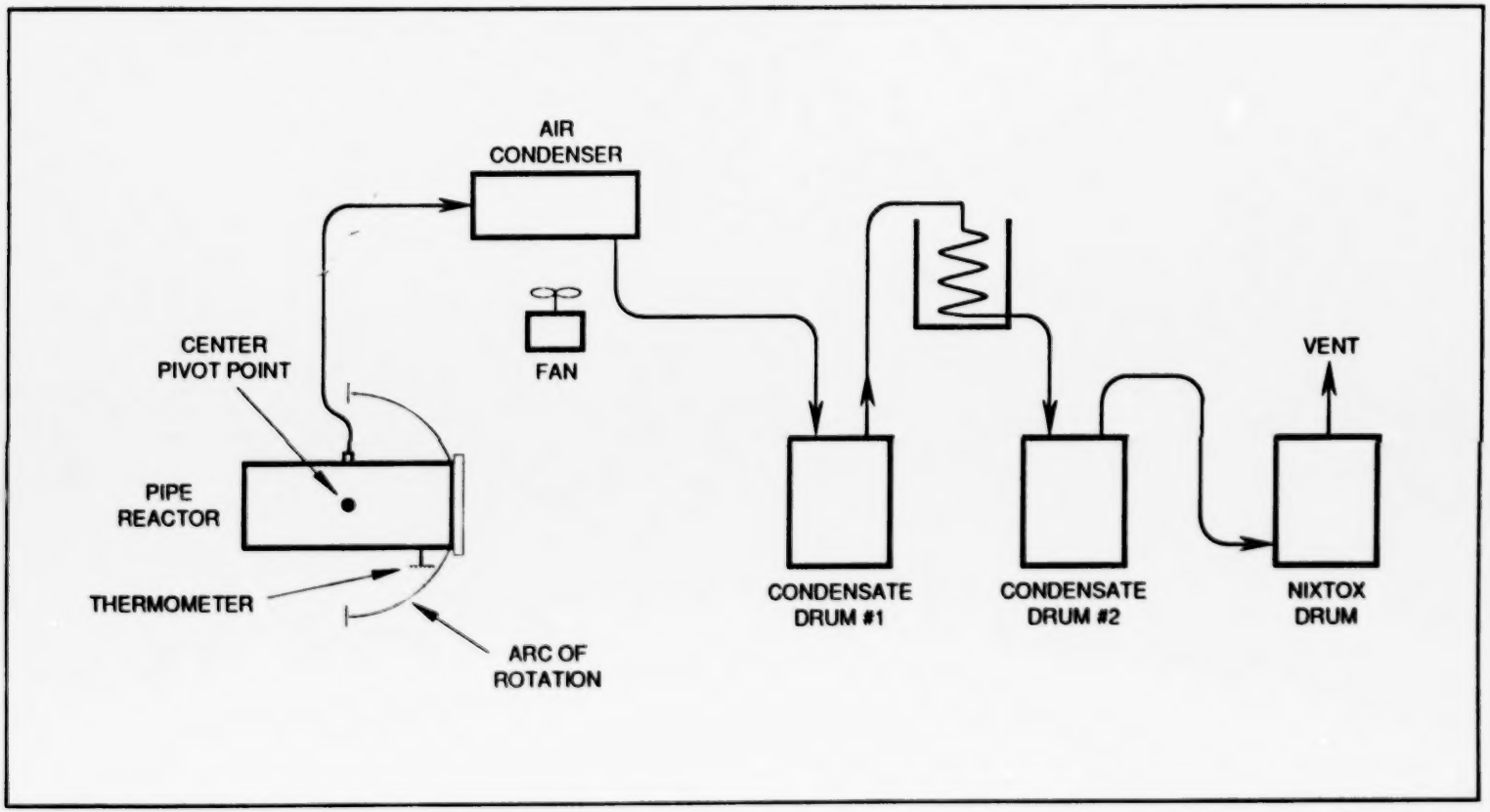


Figure 2. Equipment arrangement for the Moreau Test.

Altogether four runs were made with this reactor. Tables 2 and 3 list the reactor contents for each of these runs.

During this early field demonstration, an attempt was also made to collect and reuse the reagent. At the conclusion of each run, the reactor was allowed to cool to ambient temperature overnight, and 6 to 9 lb of water was added to dilute the reaction mixture and to promote settling of the soil so the supernatant liquid could be drained off. During these runs, the cooled soil/reagent mixture formed a gelatinous mass containing a significant amount of free liquid, which was recovered. Table 4 presents the liquid-handling and reagent-recovery aspects of these four runs. The moisture content of the soil itself ranged from 14 to 18 percent because heavy rains occurred prior to soil treatment. In addition, the water collected as condensate (see Table 3) came not only from the soil, but also from the 45 percent KOH that was added, as well as that formed by the reagent.

As shown in Table 4, the reagent recovery, based on the total amount of liquid added (neglecting the soil moisture) and the total amount of liquid drained at the end of the cycle, averaged 61 percent and ranged from 47 to 68 percent. This unexpectedly high level of reagent recovery was possible because, during the reaction, the soil formed into a porous gel-like material from which the reagent drained freely. During the draining, some grit passed through the screens and quickly settled at the bottom of the bucket; however, the drained material was generally low in total suspended solids (TSS), which ranged from 1092 to 1688 ppm. The percentage reagent recovery and the reagent TSS are also shown in Table 4.

The temperature and pressure of the reactor, which were recorded every 15 minutes during each of the four runs, did not exceed 160°C and 30 psig, respectively.

The reactor was continuously vented during Runs 1 and 2. It was also rotated through an arc of 180 degrees for each cycle. During Runs 3 and 4, a nitrogen blanket was placed over the reaction by running nitrogen through the line that was normally used to drain the reactor. The valve in the vent line was then closed and periodically cracked to control the pressure. The temperature was controlled by turning the electric heaters on and off and by removing portions of the insulation. Thermometer readings above 150°C were extrapolations because the thermometer only read to 150°C.

During each run, the reactor was sampled when the temperature reached 150°C. It was then sampled periodically until a sample showed a low PCB concentration, at which point, it was shut down.

During the third and part of the fourth run, the reactor was rotated through an arc of 90 degrees. At the end of the third run, the reactor was stopped and pounded on the end with a rubber mallet. When struck, material could be heard falling from the walls to the bottom of the reactor, which indicated that rotating the reactor at only 90 degrees allowed material to build up on the walls.

TABLE 2. MATERIAL CHARGED TO REACTOR
(pounds)

Run	Soil	Sulfolane	DMSO	TMH	PEG-400	45% KOH	Total liquid
1	33.5	0.0	11.0	5.5	5.5	11.0	32.9
2	32.5	0.0	10.0	5.0	5.0	10.0	30.0
3	34.0	0.0	12.0	6.0	6.0	12.0	35.9
4	39.0	15.0	0.0	7.5	7.5	13.0	43.0

TABLE 3. DETAILED REACTOR CONTENTS

Component	Run 1	Run 2	Run 3	Run 4
Soil, % water	14.51	14.21	14.53	18.34
Dry soil, lb	28.64	27.88	29.06	31.85
Dry KOH, lb	7.15	6.50	7.80	8.45
Water, lb	12.01	11.12	12.74	15.60
Organics, lb	22.00	20.00	24.00	30.00

TABLE 4. REAGENT RECOVERED

Run	Water, lb			Reagent drained, lb	Recovery, %	Reagent TSS,[a] ppm
	Initial	Boiled off	Added back			
1	12.01	10	6.5	15.5	47	1216
2	11.12	6.5	6.5	18.5	62	1544
3	12.74	6.5	8.5	24.5	68	1092
4	15.6	7	8.5	29.0	67	1688

[a] TSS, total suspended solids.

Table 5 shows the initial and final weights after each reaction of the two condensate collection drums and the Nixtox drum (activated carbon). No material was collected in the second condensate drum or the Nixtox drum. All the condensate was apparently condensed in the air cooler and came out in the first drum. Whereas Run 1 results show that the Nixtox drum gained about 3 pounds, the absence of a positive weight change in the next three runs indicates that this first run represents a possible measurement error.

TABLE 5. INITIAL AND FINAL WEIGHTS
(lb)

	Condensate Drum 1		Condensate Drum 2		Nixtox Drum	
Run	Initial	Final	Initial	Final	Initial	Final
1	50.0	60.0	50.5	51.0	286.5	289.5
2	51.0	57.5	51.0	51.0	289.5	287.0
3	51.0	57.5	51.0	51.0	287.0	287.0
4	52.0	59.0	51.0	51.0	287.0	286.5

ANALYTICAL PROCEDURES AND RESULTS

The analytical method used for the PCB analyses was developed from EPA-600/4-81-045, "The Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils". PCBs were extracted from soil and reagent with methanol and hexane. The samples were analyzed by the electron capture gas chromatography (EC/GC) method. The concentration of PCB in samples was calculated according to the procedure in Webb and McCall.

The PCB analyses for the pilot-scale demonstration at Moreau were performed on site. The results are shown in Table 6. The analyses were performed immediately after the samples were taken out of the reactor. Both soil and reagent were analyzed. The reduction in PCB concentration in the soil ranged from 93.9 to 99.8 percent and averaged 98.3 percent. In Run 1, the sample obtained after 4 hours showed a PCB level of 0.7 ppm, whereas the 8-hour sample showed a PCB level of 8.08 ppm. This increase in the PCB concentration may have been due to analytical error. The PCB reduction in the remaining three runs (Runs 2 through 4) was greater than 99 percent. PCBs were also found in the reagents and generally the PCB concentration in the reagent was higher than that of the soil, thus postulating that extraction of the PCB from the soil into the alkoxide phase was progressing but that chemical dechlorination was not yet complete. The PCB concentration of the reagent was 0.16 ppm in Run 1, 74.8 ppm in Run 2, 72.4 ppm in Run 3, and 4.25 ppm in Run 4.

The results of this pilot-scale demonstration indicate that the KPEG process can significantly reduce the PCB concentration in this soil. The temperature and pressure of the reaction were controllable and not subject to

unexpected excursions to unsafe levels. An appreciable quantity of the reagent was recovered from the soil during this evaluation. Although measurable quantities of PCBs were detected in the reagent during treatment, these levels were found to decrease (as expected) as the duration of the reaction increased.

TABLE 6. PCB CONCENTRATIONS MEASURED AT THE SITE
(ppm)

Hours into run	Run 1		Run 2		Run 3		Run 4	
	Soil	Reagent	Soil	Reagent	Soil	Reagent	Soil	Reagent
0	133		779		7013		206	
3							20	
3.5							19.7	157
4	0.7	0.8	50.6	74.8	228			
4.5								
5								
5.5							6.6	
6					23.5	138		
7			1.62					
7.5								
8	8.08	0.16						
8.5								
9					12.4	72.4	1.09	4.25
Condensate		1.46					26.05	

The reactor performed adequately; however, the possibility of this type of reactor being used for scale-up is clearly limited. Although the reagent formulation used to treat the soil seemed to yield promising results, disposal of the treated soil would present a problem unless residual DMSO was removed by washing. Washing soils and subsequently separating the particulate from the wash water are formidable tasks.

SECTION 4

FIELD-SCALE DEMONSTRATION OF KPEG PROCESS IN GUAM, U.S.A.

SITE DESCRIPTION

In a joint venture between the U.S. EPA/RREL and Naval Civil Engineering Laboratory (NCEL), a U.S. Navy site was selected for the field-scale demonstration of the KPEG chemical dechlorination system. The U.S. Navy Public Works Center (USN PWC) on the Island of Guam, U.S.A. was selected when analytical results of the collected soil samples indicated an average PCB concentrations of 2500 ppm with "hot-spots" as high as 45,860 ppm (4.58 percent). Soil contamination found mainly in a nearby storm drainage ditch resulted from leaks from a transformer rework building which had been used as early as World War II. The waste PCB oil that was stored outside leaked and was carried by surface runoff into the ground and drainage ditch.

Figure 3 indicates the sampling dates, locations, depths, and the PCB concentrations found in samples collected at or near the site. The transformer rework building alluded to above is shown as Building 3009. As Figure 3 shows, these soils contain sufficient PCB contamination to warrant concern.

SOIL PREPARATION FOR TREATMENT

Soil Excavation

Using an excavator and dressed in Level C protective clothing, USN PWC personnel excavated contaminated soil from the ditch before the PEI crew arrived. A 12.2-m by 18.3-m (40-ft by 60-ft) metal building was erected on the concrete pad and provided with a heavy rubber liner. The purpose of this building was to store the excavated contaminated soil. A payloader transferred the excavated soil to the metal building for temporary storage. In addition to the metal building, Navy personnel also built a 30.5-m by 30.5-m (100-ft by 100-ft) concrete pad for installation of the field-scale KPEG treatment system. Figure 4 shows the location of the concrete pad and metal building.

Soil Screening

The Navy purchased a diesel-operated mechanical screening device and brought it on the site. The interchangeable screens were adjusted so that

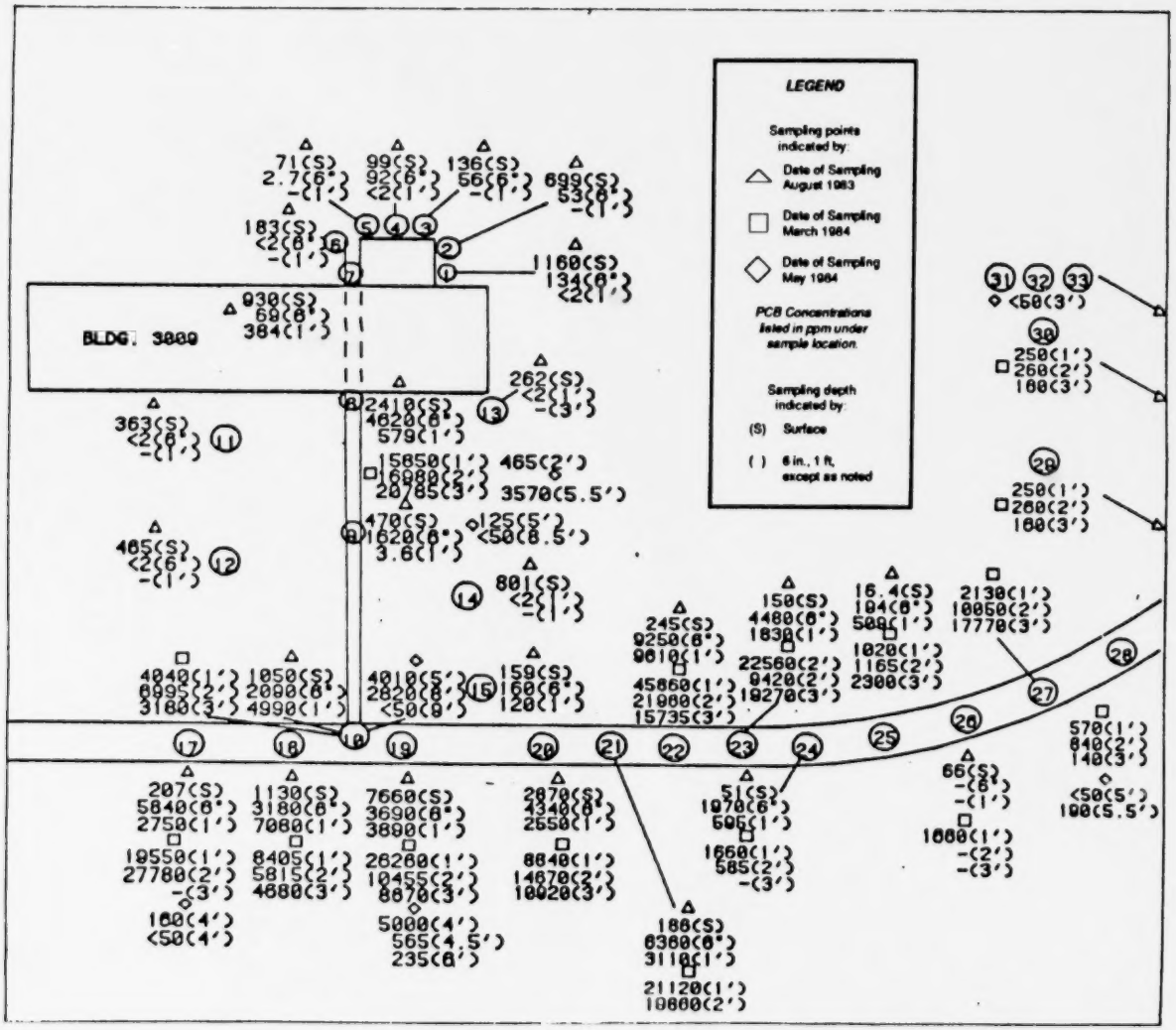

LEGEND
Sampling points indicated by:
Date of Sampling August 1983
Date of Sampling March 1984
Date of Sampling May 1984
PCB Concentrations listed in ppm under sample location.
Sampling depth indicated by:
(S) Surface
( ) 6 in., 1 ft, except as noted
BLDG. 3009

Figure 3. PCB concentration distribution (ppm), U.S. Naval Station, Public Works Center, Guam.

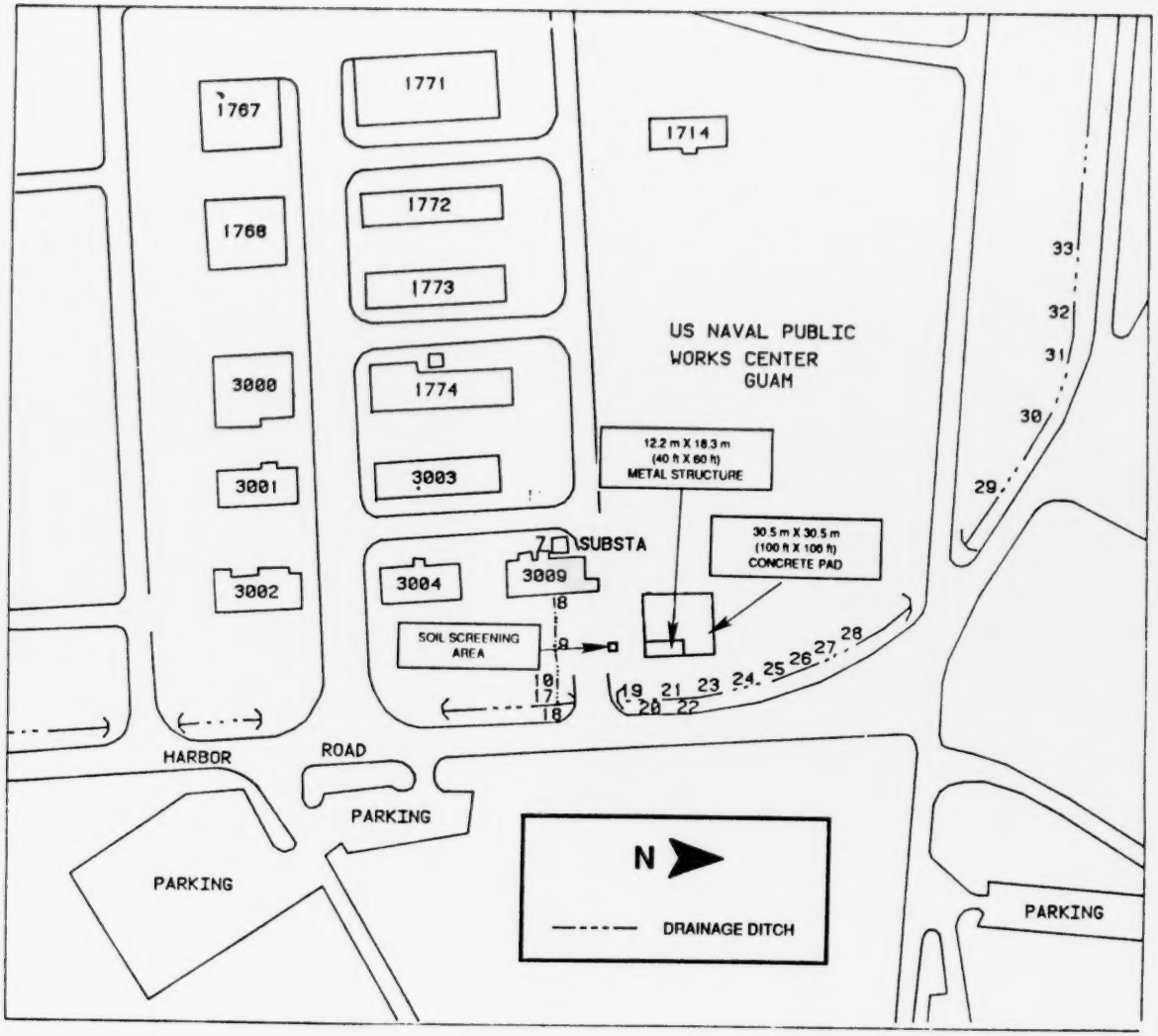


Figure 4. Storage of PCB-contaminated soil and location of field-scale KPEG demonstration.

soil and particles 1/2 inch and larger would be rejected. Soil and particles smaller than 1/2 inch passed through the screens and were collected separately.

Sizing the soil and particulates to 1/2 inch was not an arbitrary decision. The maximum size of 1/2 inch was established by the mixer manufacturer. After an evaluation of the mixing and heating efficiencies of various mixers and manufacturers, the unit offering the greatest combined mixing and heat transfer efficiency was selected. The selected mixer was designed with a shaft along the center axis of the mixing cylinder. Arms protruding radially from the shaft supported plows used for mixing and fluidizing the mixer contents. According to the selected mixer manufacturer, the maximum tolerance permitted between the plow and cylinder wall (without substantially sacrificing mixing and heat-transfer efficiency) was 5/8 inch. To avoid the jamming of particulates between the plow and cylinder wall, the mixer manufacturer established a maximum particle size of 1/2 inch.

The procedures used for screening the excavated soil deviated slightly from the screen manufacturer's written procedures because the screening operation was performed outside and dust control was of primary concern. A payloader transported a load of soil from the metal building and placed it on the top screen of the screening mechanism. The entire screen was then covered with plastic to reduce dust emissions. When turned on, the screens, which were mounted on springs, would vibrate and particles small enough to pass through the screens would be collected. Particles that could not pass through the screens would vibrate off of the slanted screen and be collected in the oversize-material pile. The entire 15.3 m^3 (20 yd^3) of soil was screened in this manner.

Storage of Screened Soil

The screened soil was returned to the metal building, where it was stockpiled separately. Approximately 11.5 m^3 (15 yd^3) of the 15.3 m^3 (20 yd^3) of soil passed through the 1/2-inch screen and was ready for KPEG treatment. The remaining 3.8 m^3 (5 yd^3) was classified as oversize and was stockpiled separately for subsequent special processing.

LABORATORY STUDIES

Results of the Moreau, New York, pilot-scale KPEG demonstration indicated that the treatment technology was viable for the dechlorination of PCBs and possibly other haloorganic compounds and that scale-up was possible. Although information collected during the Moreau demonstration was not comprehensive enough to provide full support and the detail needed for the field-scale scale-up, it was sufficient for system design work to begin while laboratory work was being performed on site-specific soil in Guam.

Laboratory studies with samples of contaminated soil from Guam were performed to determine the potential for KPEG treatment of the soil, to determine the reagent formulation and other operating parameters, and to

determine whether DMSO and TMH could be removed from the reagent. Laboratory studies were performed in Cincinnati, Ohio, by U.S.EPA-RREL and PEI personnel concurrently with PEI's initial system design. Laboratory results and conditions were factored into PEI's design of the field-scale system.

Removal of DMSO from the formulation was appropriate from a health and safety standpoint. The excellent solvent characteristics of DMSO, coupled with the known rapid rate of skin penetration posed serious concerns for workers in the presence of compounds such as PCBs and PCDDs. In addition, while using noncontaminated soil during an evaluation of a prototype mixer in a manufacturer's facility, a fire occurred when air accidently entered the mixer through a leaky seal and contacted with the reactor contents (DMSO, TMH, PEG-400 and KOH). DMSO or TMH vapors were suspected of having been ignited either spontaneously or in conjunction with static electricity. In any case, laboratory studies led to the finding that DMSO and TMH were not needed to accomplish degradation of the PCBs present in Guam soil.

FIELD-SCALE KPEG SYSTEM DESIGN

On the basis of the results obtained from the laboratory studies and from the pilot-scale demonstration at the Moreau, New York, site, PEI engineers designed the KPEG treatment system for the demonstration at the Guam site. A simplified mechanical flow diagram of the KPEG system is shown in Figure 5. The diagram illustrates that the mixer was the primary component of the system where the chemical dechlorination process occurred and was supported by ancillary equipment to make the system functional. An extensive pipe network was required for interconnecting the ancillary equipment to each other and to the mixer. To reduce the complexity of the piping network, a centralized pipe rack for all interconnecting piping was designed. A layout of the Guam site, as shown in Figure 6, indicates the locations of all ancillary equipment. The majority of the auxiliary system was located outside the exclusion zone for easy access without requiring Level C protection. Only equipment contacting the soil in its contaminated state and the equipment that had to be immediately adjacent to the mixer were in the exclusion zone. A detailed mechanical flow diagram of the system and the detailed layout of the site plan are shown in Figures 7 and 8, respectively.

DESCRIPTION OF PROCESS EQUIPMENT

The principal components of the KPEG treatment system used in the Guam field test are described in the following subsections.

Mixer (KPEG Reactor)

The selected mixer was designed with a total capacity of 793 gallons (106 cubic feet) and a working capacity of 490 gallons (65 ft^3). The mixer was equipped with a 75-hp motor and gear box capable of providing mixer shaft speeds of 30 and 60 rpm. All potentially wetted parts were constructed of

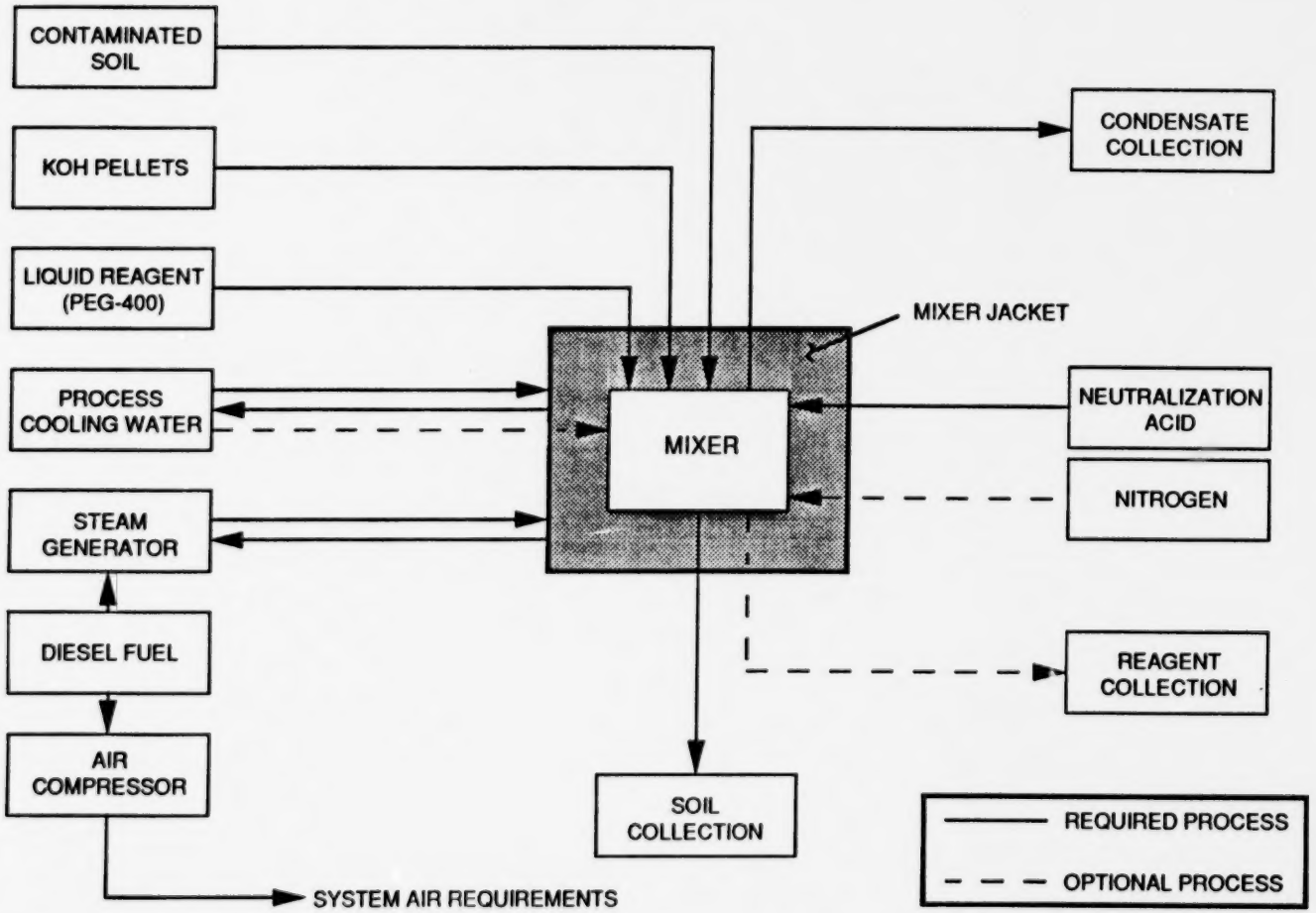


Figure 5. Mechanical flow diagram of KPEG field-scale treatment system.

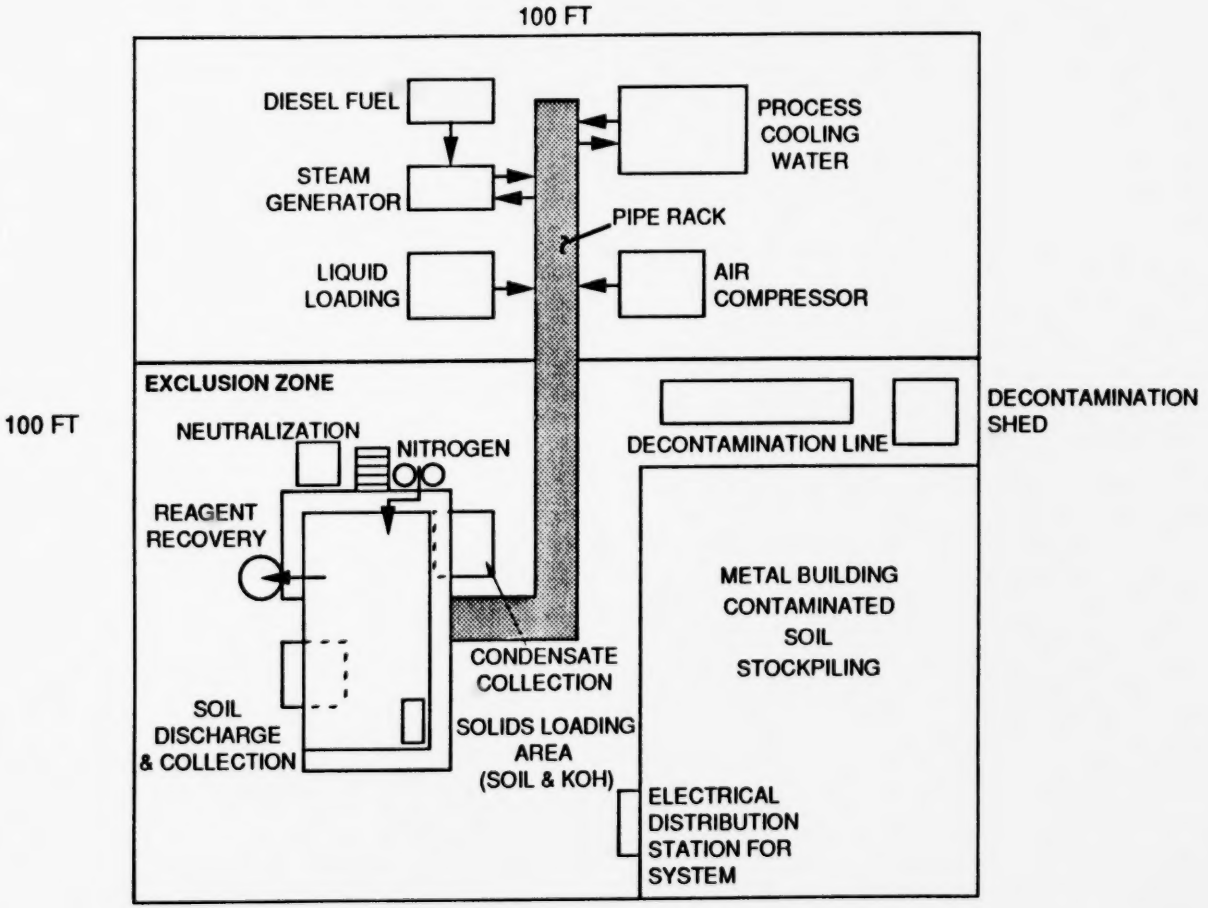


Figure 6. Site plan for KPEG field-scale treatment system.

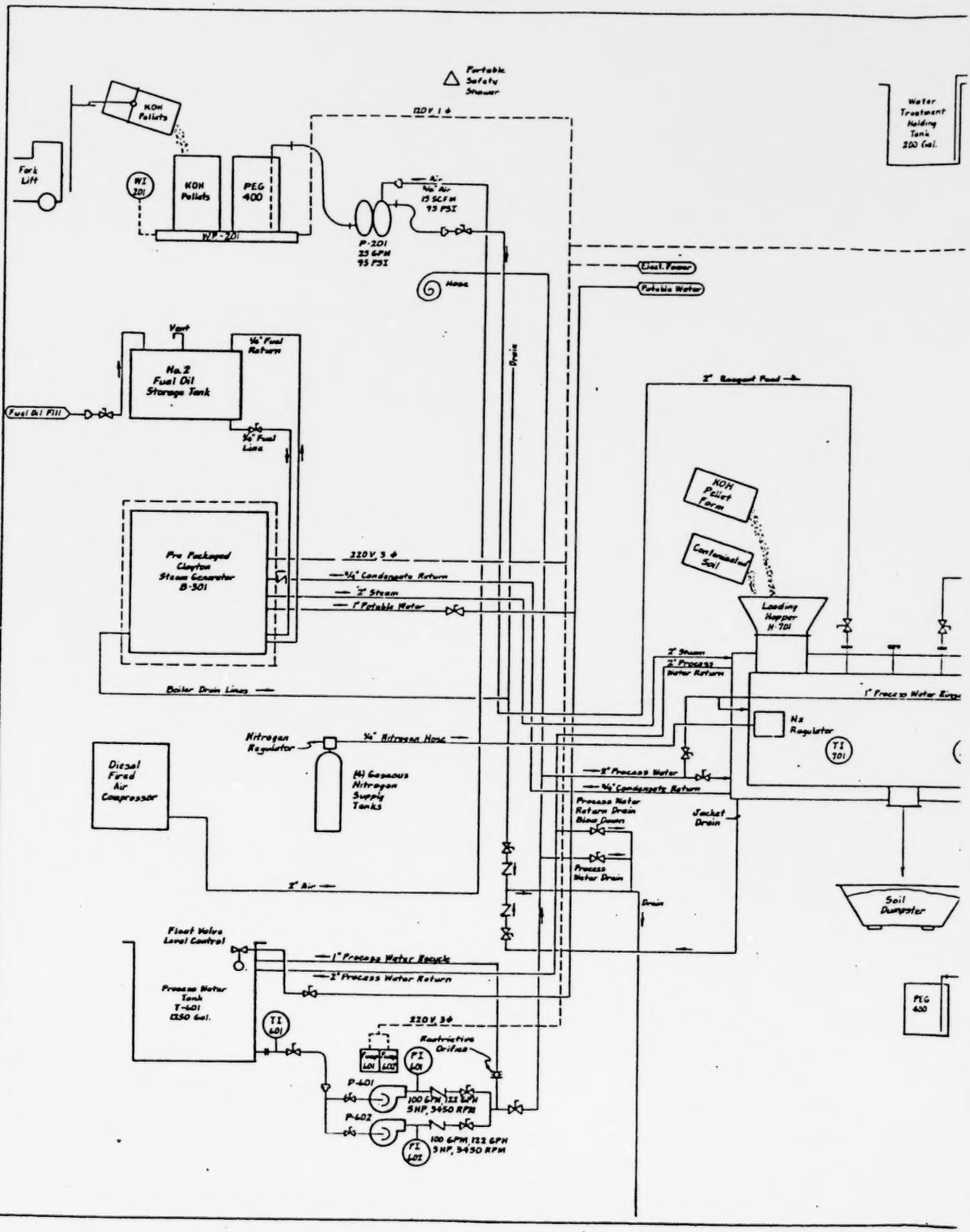
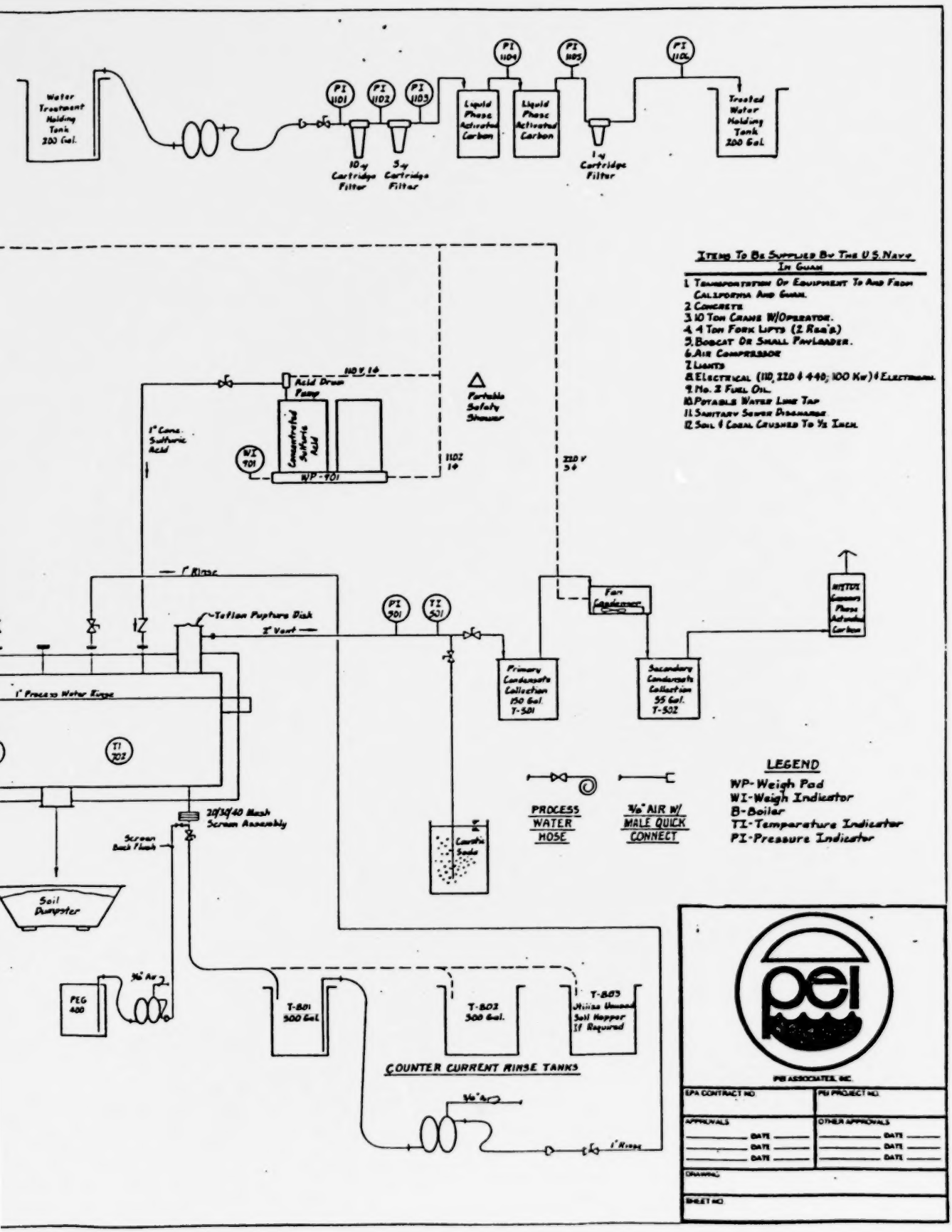


Figure 7 Mechanical flow diagram of KPEG –



→ field-scale treatment system.

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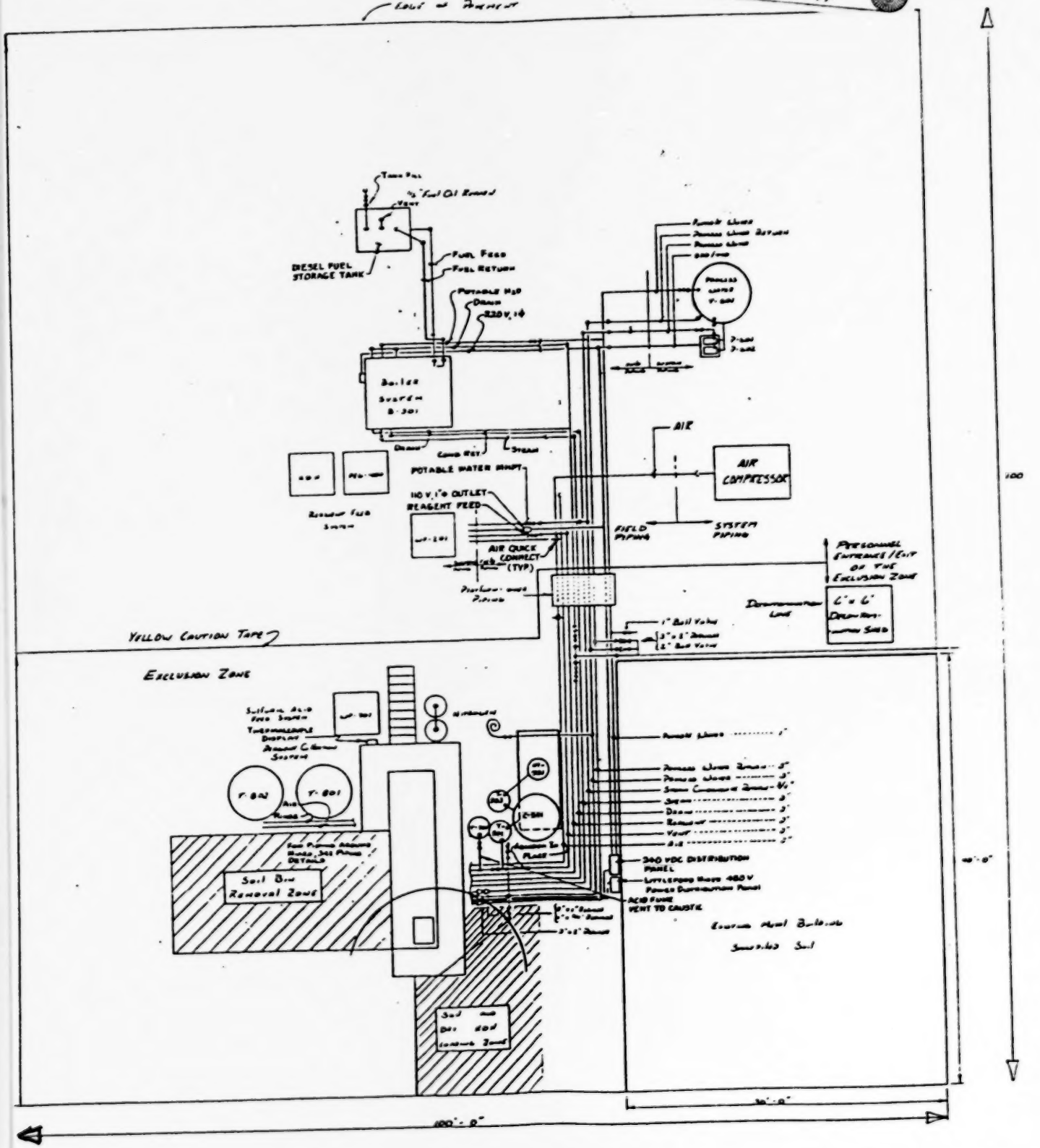

Figure 8 Site plan for KPEG field-scale treatment system.

316 stainless steel to prevent corrosion from chemical attack. The mixer had an 8-inch-diameter shaft that ran the length of the mixing cylinder and was supported at each end by the positive-flow nitrogen purged seals. Extending radially from the shaft were arms with V-shaped plows that maintained a 5/8-inch tolerance from the wall. The manufacturer recommended that a maximum tolerance of 5/8-inch between the plow and wall be maintained to prevent the creation of a dead zone where caking could occur, which would substantially reduce mixing and heating efficiencies. This 5/8-inch tolerance also established the maximum allowable particulate size of 1/2 inch to assure that no particulate jamming occurred between the plow and wall.

Figure 9 presents a diagram of the selected mixer and designed features. Three 2-inch and one 1-inch flanged liquid charge ports were located along the top of the mixer for the addition of PEG-400 and sulfuric acid. One 1-inch charge port was provided at each headwall of the mixer for the addition of water. A 12-inch Teflon-coated rupture disc was provided at the top of the mixer as a precautionary measure against overpressurization of the mixer. A 2-inch vent was also provided, which led to the condensate collection system.

Solids were charged into the mixer through a 20- by 24-inch rectangular flange. A 16-inch flanged screen assembly with a 2-inch drain was provided at the bottom of the mixer for draining the reagent after treatment. Treated soils were discharged from the mixer through an 8-inch air-operated ball valve located on the bottom center of the mixer.

The mixer cylinder was provided with a steam jacket rated at 80 psi (156°C). Manifolds were provided across the top and bottom of the steam jacket. During heating, steam entered the top manifold, traversed downward through the jacket, and exited the bottom manifold. The manifolds were designed to serve as part of the cooling system as well. Rearrangement of the valves allowed for the upflow of cooling water through the jacket.

Platform

The entire mixer/motor assembly was mounted on a platform so the discharge port would be sufficiently elevated to allow for placement of a soil collection hopper underneath the mixer for discharge. For access, the platform was designed with catwalks around the mixer. These catwalks folded down for transport in order that legal road widths were not exceeded. The platform was designed with an integrally mounted jib crane to lift drums of soil and dry KOH for charging into the mixer.

Liquid Reagent Loading System

The liquid-reagent loading system consisted of a pallet scale and air-operated diaphragm pump. The PEG-400 was placed on the scale and tared. The positive-displacement pump was used to charge proper quantities of PEG-400 into the mixer.

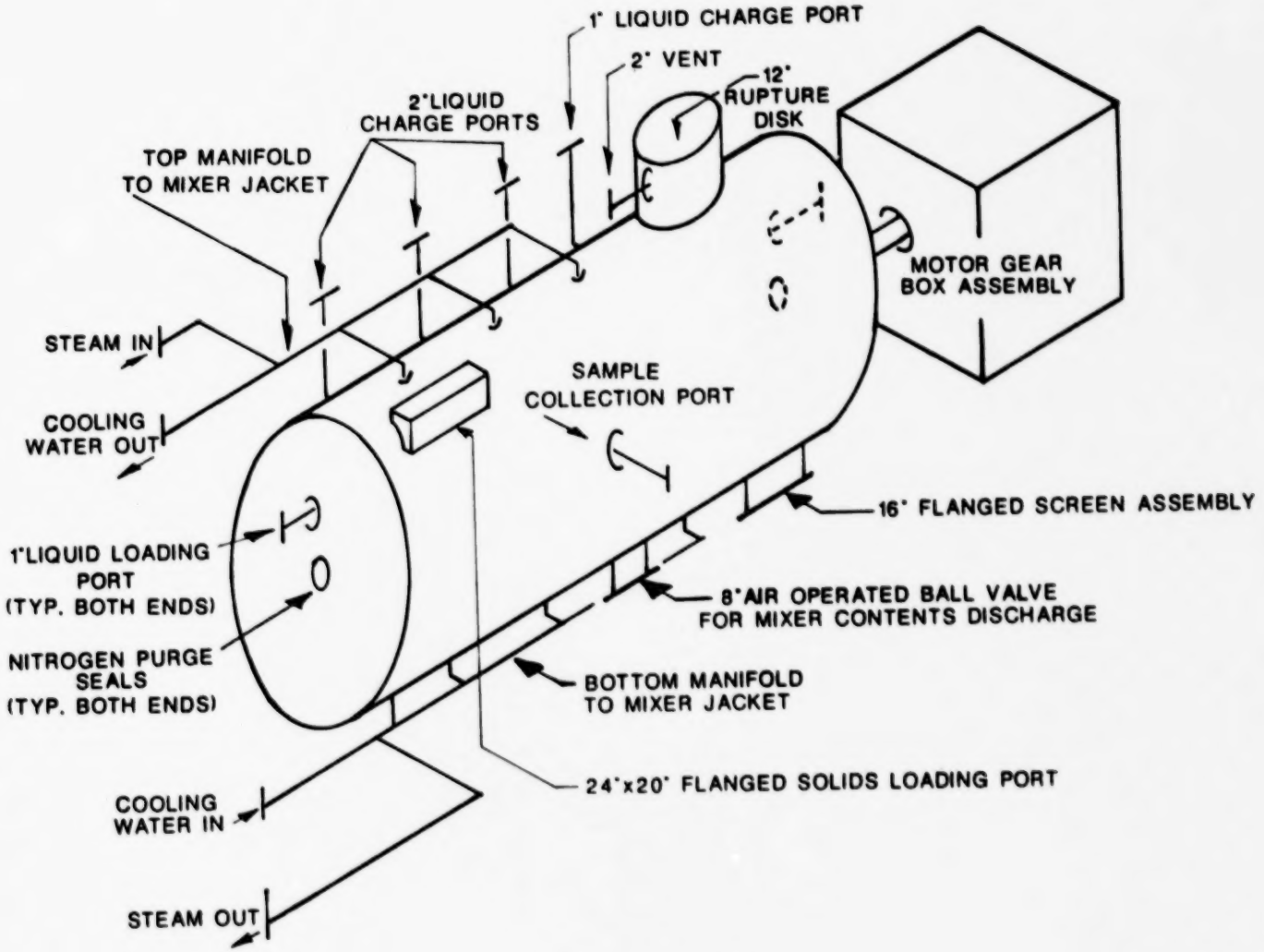
1" LIQUID CHARGE PORT
2" VENT
12" RUPTURE DISK
2"LIQUID CHARGE PORTS
TOP MANIFOLD TO MIXER JACKET
MOTOR GEAR BOX ASSEMBLY
STEAM IN
COOLING WATER OUT
SAMPLE COLLECTION PORT
16" FLANGED SCREEN ASSEMBLY
1"LIQUID LOADING PORT (TYP. BOTH ENDS)
8"AIR OPERATED BALL VALVE FOR MIXER CONTENTS DISCHARGE
NITROGEN PURGE SEALS (TYP. BOTH ENDS)
BOTTOM MANIFOLD TO MIXER JACKET
24"x20" FLANGED SOLIDS LOADING PORT
COOLING WATER IN
STEAM OUT

Figure 9. Field-scale KPEG mixer and design features.

Heating System

The heating system was a leased package steam-generating plant. Design calculations based on approximated soil and moisture contents and reagents indicated that a 600-lb/h, 80-psi unit was required to heat the mixer contents from ambient temperature to 150°C within a 4-hour time frame. Greater steam pressure (higher temperature) could not be used because the steam jacket rating on the mixer was 80 psi. The mixer steam manifold included a steam-pressure-relief valve specified at 80 psi.

Nitrogen System

The nitrogen system provided in the design was part of the safety consideration in the event DMSO and TMH were not removable from the KPEG formulation. The nitrogen system consisted of a pressure regulator and flow controller. Nitrogen was purged into the mixer through the seals to provide a nitrogen atmosphere inside the mixer. The removal of DMSO meant the nitrogen was no longer required, and its use was discontinued when it was learned the seals would not become damaged without the nitrogen flow.

Condensate Collection System

The design of the condensate collection system was similar to that used in the pilot-scale demonstration in Moreau. The field-scale mixer was vented to a steel knockout tank, which was provided to collect soil particulates which may have been thrown into the vent line from the vigorous mixing action. The knockout tank vented to a fan condenser where 9600 scfm ambient air was blown over the condenser coils. The fan condenser drained into a condensate collection tank, which was originally vented into a secondary condenser consisting of a copper coil submerged in an ice bath. Restrictions in the line size at the ice condenser created back pressure on the system, which necessitated its removal. The condensate collection tank was then vented directly to the activated-carbon column for collection of any remaining volatilized organics.

Process Cooling Water System

The hot treated soil contained within the mixing cylinder was cooled before further processing. The process cooling water system consisted of a 1250-gallon water tank and centrifugal pump assembly. Piping on the mixer manifolds was arranged to allow cooling water to flow up through the mixer jacket and to return to the water tank.

Reagent Collection System

The reagent collection system consisted of the two liquid-charge ports on the mixer headwalls, the 16-inch screened flange and drain on the bottom of the mixer, and two 500-gallon steel tanks immediately adjacent to the mixer. Ideally, following treatment, the soil was washed with water and the reagent allowed to drain through the screen into the 500-gallon tanks for potential reuse.

Neutralization System

The neutralization system consisted of a pallet scale, sulfuric acid, and drum pump. A stoichiometric quantity of sulfuric acid was pumped into the mixer to neutralize the known quantity of KOH. The high calcium carbonate ($CaCO_3$) content of the soil required additional acid to reduce the pH of the mixer contents to a range of 6 to 9.

PREASSEMBLY AND TRANSPORT OF KPEG SYSTEM TO GUAM

Preassembly of System in Cincinnati Warehouse

Upon completion and approval of the system design, the necessary equipment was ordered. When the equipment arrived in a Cincinnati warehouse, the PEI crew assembled the system in the warehouse to the extent time limits permitted. Assembly was approximately 90 percent complete before disassembly was initiated. Disassembly was performed systematically, and each piece was marked with appropriate field-erection notes. All of the equipment was crated and loaded onto tractor-trailers for transport to Port Hueneme, California.

Transport to Guam

Three flatbed trailers packed with KPEG treatment equipment were transported to Port Hueneme, California, where it was unloaded and placed with the reagent chemicals that had been received from a local chemical supplier.

The equipment and chemicals were loaded onto a U.S. Navy cargo ship and shipped to the Island of Guam, USA. Upon arrival in Guam, USN personnel transferred the equipment and chemicals onto flatbed trailers and transported them to materials receiving, where most were again off-loaded. After passing through materials receiving, the equipment and chemicals were again loaded onto flatbed trailers, transported to the site, and off-loaded. The crated systems were moved no less than nine times before being reassembled. This movement and relocation to a site half-way around the world successfully demonstrated the portability of the field-scale KPEG system and leads one to believe that larger systems can also be made portable by the use of trailers dedicated to and integrally designed into the system.

System Assembly in Guam

Upon arrival of the KPEG equipment and PEI crew at the Guam site, the crew reassembled the system with the assistance of USN PWC personnel and equipment. The first two pieces of equipment placed on site were the mixer platform and the mixer onto the platform. A U.S. Navy crane, operator and rigger were used to position the platform and mixer. When the mixer was in place, the pipe racks were laid out in the proper locations and coupled together. Other major pieces of equipment (the reagent recovery tanks, sulfuric acid systems, condensate collection system, reagent weighing and pumping system, nitrogen purge system, steam generator, diesel fuel supply

system, process cooling water system, air supply system, and process-water drain tanks) were then positioned. All connecting piping between the individual systems, the pipe rack, and the mixer were plumbed in. The pipes were pressurized to detect any leaks and flushed to remove any foreign debris (e.g., metal shavings, cutting oil, or corrosion) that may have accumulated.

All electrical wiring was connected. The USN PWC provided the necessary personnel and equipment to provide electrical service from the substation transformer to the system electrical distribution panels. The electrical system was first checked out with a volt meter at each junction box and equipment connection point. All equipment was then electrically bumped to assure proper rotation and operation.

PROCESS IMPLEMENTATION

Transport of Soil

Because the loading of the soil was not automated, a safe and convenient method was devised for efficient manual moving of 1.5 m^3 (2 yd^3) of soil to a height of approximately 3.7 m (12 ft). Six 55-gallon drums were placed on a metal pallet that could be handled by forklift and were positioned in the metal building next to the stockpiled soil awaiting KPEG treatment. Two persons dressed in Level C protective clothing entered the building and shoveled the soil into the drums. When all of the drums were filled, the lids were placed on the drums and the pallet was swept off.

Determination of Soil Weight

The six 55-gallon drums of soil were relocated to the scale, where a small forklift with a drum-lifting attachment placed the tared drums full of soil on the scale. Weighing several drums during the first batch indicated that the weight of soil per drum was fairly consistent. As a time-saving measure, it was decided that no further weighing would be performed and that 55-gallon drums filled to 90 percent capacity (50 gallons) contained 257 kg (567 lb) of soil.

Pretreatment Soil Sample Collection

A grab sample was collected from each of the six drums of soil. The samples were placed in a container and mixed thoroughly to provide a homogeneous sample. Samples from this composite were placed in sample jars and properly labeled. The excess composite was returned to the stockpile.

Loading the Mixer

The soil-loading port was approximately 3.7 m (12 ft) above the ground when the soil-loading hopper was in place. A manual job crane was integrally designed into the platform to hoist the 257 kg (567 lb) drums of soil to the loading hopper. Unfortunately, the support column for the crane was lost

during shipment between Cincinnati and Guam. To facilitate loading the soil into the mixer, the U.S. Navy provided a crane and operator to hoist the drums of soil.

Three persons in Level C personal protective clothing were required to load the soil into the mixer. Two of them were on the platform at the soil-loading port, and the third was on the ground. As the flange cover was removed from the soil-loading port, the soil-loading hopper was rigged to the crane and hoisted to the loading port. The loading hopper was bolted to the mixer flange to hold it in place.

With a wrap-around drum lifter, the crane hoisted and positioned the drum of soil over the loading hopper. Using the manual chainfall on the drum lifter, the two persons on the platform dumped the drum contents into the mixer. This procedure was followed until all six drums of soil had been loaded into the mixer. After the loading of every second drum of soil, the mixer was turned on for a short time to disperse the soil evenly throughout the mixing cylinder.

Loading Reagent Into Mixer

After the soil was loaded into the mixer, the components of the KPEG formulation were added. Initially, they were added at a rate of 1.0 mole of KOH and 1.0 mole of PEG-400 to provide a 1/2:1 weight relationship of mixed KPEG to soil. The formulation was then adjusted, and a KPEG formulation of 1.3 moles of KOH and 1.0 mole of PEG-400 was selected. The components of the KPEG mixture were still added to the soil to provide a 1/2:1 KPEG-to-soil weight relationship.

The calculations used to determine the quantities of KOH and PEG-400 required per batch did not prove to be difficult. Based on a ratio of 1.3 mole KOH to 1.0 mole PEG-400 for the KPEG formulation, the KOH and PEG-400 quantities were determined by the following calculations:
Soil:

$$6 \text{ drums} \times 567 \text{ lb/drum} = 3402 \text{ lb soil} \Rightarrow 3400 \text{ lb soil} \quad (3)$$

Molecular weights:

PEG-400: 399 [400 less An H^+ (M.W. = 1)]
K: 39 (M.W. KOH = 56)

KPEG: 438

Quantity of KPEG desired:

$$\frac{3400 \text{ lb soil}}{1} \times \frac{1/2 \text{ lb KPEG}}{1 \text{ lb soil}} = 1700 \text{ lb KPEG} \quad (4)$$

$$\frac{1700 \text{ lb KPEG}}{1} \times \frac{1 \text{ mole KPEG}}{438 \text{ lb KPEG}} = 3.88 \text{ moles KPEG} \quad (5)$$

PEG 400:

$$\frac{3.88 \text{ moles KPEG}}{1} \times \frac{1 \text{ mole PEG}}{1 \text{ mole KPEG}} \times \frac{400 \text{ lb PEG}}{1 \text{ mole PEG}} = 1553 \text{ lb PEG-400} \quad (6)$$

$$=>1555 \text{ lb PEG-400}$$

KOH:

$$\frac{3.88 \text{ moles KPEG}}{1} \times \frac{1.3 \text{ mole KOH}}{1.0 \text{ mole KPEG}} \times \frac{56 \text{ lb KOH}}{1 \text{ mole KOH}} = 282 \text{ lb KOH} \quad (7)$$

$$=>285 \text{ lb KOH}$$

Equations 6 and 7 represent the quantities of PEG-400 and KOH pellets, respectively, that were required for each six drums of soil, 1542 kg (3400 lb), placed in the mixer for each batch of KPEG treatment.

KOH Loading--

After all six of the drums of soil were loaded into the mixer, preweighed KOH pellets were also loaded into the mixer in the same manner. The soil-loading hopper was then removed, a new nonasbestos-type gasket was installed, and the flange cover was bolted back into place. (A new nonasbestos-type gasket was required for each batch because the original material could not retain its elasticity and was therefore unable to provide a satisfactory seal following one use.)

PEG-400 Loading--

When the flange was back in place, the mixer was turned on to low speed (30 rpm). The PEG-400 was loaded by pumping. A pallet of PEG-400 was placed on the scale and tared, and an air-operated diaphragm pump was used to pump the proper quantity of PEG-400 into the mixer. The mixer was then fully charged and ready for treatment.

OPERATION

Mixing

The mixer was turned on at low speed (30 rpm) while the PEG-400 was pumped into it, which allowed thorough dispersion of the PEG-400 and KOH to initiate the first reaction shown in Equation 1. When the reagents and soil were completely mixed, the mixer was turned to high speed (60 rpm), even though reactions were not yet complete. A speed of 60 rpm was sufficient to partially fluidize the mixer contents so as to provide greater efficiency for heat transfer. The vent line from the mixer to the condensate collection system was opened, and the fan condenser was turned on.

Heating

The steam-generating plant was ignited, and 80-psi (156°C) steam was circulated through the mixer jacket. The mixer contents reached 150°C in approximately 2 hours. Steam pressure was reduced to 70 psi (150°C), and the temperature and mixing were maintained for a period of 4 hours. At the completion of the 4-hour period, the steam generator and mixer were shut down. The fan condenser was turned off, and the contents were allowed to cool overnight.

Washing

During the first batch, noncontaminated soil was used to shake down the new system. Attempts were made to wash the soil with water and to drain and collect the reagent for potential reuse. Two hundred gallons of water was added to the mixer for washing and the drain on the screened flange was opened, but no drainage occurred. The screen was then back-flushed to remove any plugging, and still no draining occurred. When the top flange was opened, it was found that the soil had settled, the reagent had formed a supernatant layer, and the reagent was not filtering through the soil as it had in the Moreau pilot-scale demonstration. The supernatant was pumped out of the mixer into a 500-gallon reagent tank. The reagent was neutralized with sulfuric acid, collected by the U.S. Navy Public Works Center (USN PWC) liquid-waste hauler, and transported to the sanitary sewer treatment plant. The soil was also neutralized with sulfuric acid and discharged into a soil-collection hopper for storage. A decision was made to discontinue soil washing and reagent recovery during the field-scale demonstration. Treated soil would just be neutralized and discharged.

Neutralization

All subsequent batches of soil were immediately neutralized and discharged after cooling overnight. Additional cooling of the mixer contents was required prior to neutralization because of the strong exothermic reaction expected. Overnight cooling lowered the temperature of the mixer contents from 150° to 90°C. Additional cooling was then performed by recirculating cooling water in an upflow manner through the mixer jacket until the mixer contents were cooled to 50°C. The cooling water was kept running, and a stochiometric quantity of 200 lb of sulfuric acid was added in 20-lb increments. Because of the known high calcium carbonate content in the Guam soil, the additional sulfuric acid was believed to be required to adjust the pH to within a range of 6 to 9. Samples were collected from the sample-collection port, and the slurry pH measured. Additional 20-lb increments of sulfuric acid were added and the pH measurement process repeated until the pH was within the 6 to 9 range. A total of 280 lb of sulfuric acid per batch was required to neutralize the mixer contents.

Posttreatment Soil Samples

The collected sample found to be within the 6 to 9 pH range was also used as the posttreatment soil sample. It was distributed into the

appropriate sample jars and properly labeled. Any excess samples taken during neutralization were returned to the discharge hopper.

Mixer Discharge

With the soil-collection hopper in place and the mixer operating at low speed (30 rpm), the 8-inch, air-activated, discharge ball valve was opened from the mixer control panel. The mixing action of the mixer moved the contents to the discharge valve for removal. Mixer contents were discharged within 10 minutes. The air-activated ball valve then was closed and the mixer turned off.

STORAGE OF TREATED SOIL

The treated soil and reagents used in the treatment process were discharged into the soil-collection hoppers pending analytical results. The filled soil-collection hopper was removed from underneath the mixer discharge, and the lid was bolted on. The soil-collection hopper was then placed in an area designated for storage of treated soil. This storage area was located within the hot zone in the event of a spill of a batch from a soil-collection hopper that failed to meet the R&D permit requirements. Before PEI's final departure from the site, all soil hoppers containing KPEG-treated soil were positioned within the metal building.

SYSTEM DECONTAMINATION AND DISASSEMBLY

A high-pressure (2000 psi) water washer was leased for decontamination of the process equipment. Although no PCB and PCDD contaminants were believed to remain inside the mixing cylinder, because of the numerous KPEG treatment batches handled, the inside of the cylinder was decontaminated, and the decontamination water was collected in 55-gallon drums. The external portion of the mixer, the platform, and all other ancillary equipment in the hot zone were also decontaminated. The equipment located outside the hot zone also was rinsed with the high-pressure water washer for good measure. All decontamination water was pumped from the concrete containment area into a collection tank, and the concrete was then pressure-washed. Again, the wash water was pumped into a collection tank. All collected decontamination water was held for processing.

The KPEG treatment system was then disassembled and prepared for shipment to the U.S. mainland according to procedures indicated by EPA's Task Project Monitor. All major equipment items were disconnected from the system and staged so it would be ready for crating by U.S. Navy personnel and transport back to the U.S. mainland.

TREATMENT OF COLLECTED CONDENSATE AND DECONTAMINATION WATER

As part of the complete KPEG field-scale demonstration system, a small treatment system was provided for decontamination water. The collected

condensate and decontamination water were passed through a series of cartridge filters and activated-carbon canisters. The treated water was collected, sampled, and held until analytical results for PCB contamination were obtained. Treated waters with nondetectable PCB concentrations were either collected by the liquid transport truck and discharged into the sanitary sewer treatment plant or pumped back onto the hot zone.

ANALYTICAL PROCEDURES AND RESULTS

Analyses for PCB were performed by U.S. EPA/RREL personnel in the U.S. Navy's field laboratory at the USN PWC site (FENA Laboratory). The method used in the FENA Laboratory was an analytical procedure U.S. EPA/RREL developed for the quantitation of PCBs in soils. The method was designed to use equipment available in the Navy's FENA Laboratory on Guam. The method, which was not validated, was regarded as a field-screening method--thus the requirement for corroborative analysis by the laboratory at IT/ Knoxville. The procedure used in the FENA Laboratory in Guam entailed extracting the PCBs from the sample matrix and quantitating them in the extract with a gas chromatograph-mass spectrometer (GC/MS). This method used the DCMA* PCB standard to prepare calibration curves for use in quantitating the residual PCB congeners, as was stipulated by the R&D Permit.

The IT/Knoxville laboratory used a more vigorous procedure previously developed for EPA to analyze for PCBs. The corroborative analyses were implemented to confirm results obtained in the field laboratory. The soil samples were prepared and analyzed by gas chromatography/mass spectrometry using a modification of the method developed for EPA entitled, "Analytical Method: The Analysis of By-Product Chlorinated Biphenyls in Air, Revision 2," published on May 20, 1985. Samples were analyzed with the same frequency as specified for the quantitation in the field.

Results on the analyses of untreated Guam soil by the field laboratory and those of the corroborative analyses by IT/Knoxville differed slightly. The PCB concentrations of the untreated soil from the corroborative analyses were reported as recovery corrected. On site PCB concentrations of the untreated soil were not recovery-corrected when reported, thus justifying the consistent discrepancy between the two laboratories. Analytical results of the KPEG field-scale demonstration performed in Guam are presented in Table 7.

A review of treated-soil analytical data from both laboratory facilities indicated that the destruction of the total PCB concentration exceeded 99 percent. Unfortunately, in both sets of data, analysis of each of the congener peaks showed that a tetrachlorobiphenyl congener concentration in a portion of the treated batches was slightly above the R&D permit requirement of 2 ppm or lower per PCB peak. Additional laboratory work on the once-treated soil showed that the addition of more base (KOH) and further treatment

* Dry Color Manufacturers Association.

TABLE 7. CONCENTRATIONS OF PCBs FOUND IN PRE- AND POSTTREATMENT GUAM SOILS, CONDENSATE, AND KPEG REAGENT AND PERCENTAGE REDUCTIONS ACHIEVED FOR SOILS

Batch No.	Initial PCB Concentration in soil		Concentration of PCB in soil following treatment		Percentage reduction (using Lab No. 2 data)	Concentration of PCB's found in condensate (using Lab No. 2 data)	Concentration of PCB's found in recovered reagent (using Lab No. 2 data)
	Lab No. 1	Lab No. 2	Lab No. 1	Lab No. 2			
1	1990[a]	3276[a,b]	15.2[c]	13.9[b]	99.5757	0.831	0.01
2	1740	3828	1.99	1.01	99.9736	0.176[d]	0.14
3	1490	3406	1.45	2.86	99.9160	-[d]	0.56
4	1540	3778	1.94	3.03	99.9198	13.81	-
5	1010	3651[b]	1.91	3.31	99.9093	3.50	-
6	1410	3260[b]	4.38	7.77	99.7617	125[e]	-
7	1140	-[f]	3.48	8.56	-	-	-
8	1860	-[f]	4.94	g	-	-	-
1-R[h]	15.2	-	0.48	-	96.84[i]	ND[j] (1.0)	-
6-R	4.38	-	0.85	-	80.60	ND (1.0)	-
7-R	3.48	-	0.15	-	95.69	ND (1.0)	-
8-R	4.94	-	0.66	-	86.64	ND (1.0)	-

[a] The concentrations reported by Laboratory 2 are recovery-corrected. The initial PCB concentrations reported by Laboratory 1 are not corrected for recovery for Batches 1-8; therefore, these values are low when compared with the low-resolution recovery-corrected values obtained by Laboratory 2. These data were obtained by use of extraction and gas chromatography/mass spectrometry. Lab 1 indicates EPA Lab and Lab 2 indicates IT/Knoxville Lab.

[b] Average of duplicate analysis.

[c] Recovery corrected based on recovery of DCMA standard from Guam soil, triplicate analysis.

[d] Composite of condensate resulting from treatment of Batches 2 and 3.

[e] Average of triplicate analyses.

[f] Not analyzed.

[g] Inordinately high value was disregarded.

[h] Batches of soils that were retreated.

[i] Percent reduction from previously treated material.

[j] ND = none detected; limits of detection listed in parentheses.

reduced the tetra concentration to below 1 ppm. This work led to alteration of the KPEG reagent for the Guam soil by increasing the ratio of KOH in the mixture from 1.0 to 1.3 moles of KOH per mole of PEG-400.

The treated soil was contained in the soil-collection hoppers, which kept each treated batch separate. The batches containing greater than 2 ppm of tetra congener were loaded back into the mixer, mixed with the new KPEG formulation of 1.3 moles KOH and 1.0 mole PEG-400, and retreated. Analytical results of the retreated batches indicated the tetra congener in each batch was well below 1 ppm.

Based on the field laboratory and corroborative laboratory analyses, which used slightly varying procedures, final results on the treated Guam soil indicated that all PCB congener concentrations were below 2 ppm (as required by the R&D Permit) and the final pH was within the range of 6 to 9. As defined by the conditions of the R&D Permit, KPEG treatment of the soil was found to reduce the concentration of the PCBs in the soil to below the limits of 2 ppm per detectable PCB congener, as set by the permit.

SECTION 5

KPEG DRUM TREATMENT DEMONSTRATIONS

The preceding sections have dealt specifically with the KPEG demonstrations that have been performed to prove the efficacy of the KPEG process on larger scales and to promote the development of a full-scale system. This section describes the in situ treatment of smaller quantities of contaminated materials that may have already been placed into drums for storage. Since a large number of sites exist where contaminants have already been drummed, an in situ, drum-scale treatment process was developed to treat the contaminated material in the drum in which it was stored. This process offers an alternative to the costly and hazardous process of emptying drums of hazardous materials. Although several KPEG drum treatment demonstrations at various sites have been performed, only two are discussed in this chapter - the Bengart & Memel site in Buffalo, New York, where drummed PCB-contaminated soil was successfully dechlorinated and a site in Omaha, Nebraska, where drums of contaminated herbicide wastes containing dioxins were successfully treated. The two KPEG drum scale treatment demonstrations referred to above and described in detail below were performed by the EPA Risk Reduction Engineering Laboratory and in the case of the Bengart & Memel site by the Galson Research Corporation (GRC), Buffalo, New York.

BENGART & MEMEL SITE

Site Description

Bengart & Memel, Inc., a wholesaler of nonferrous scrap metals, was founded in 1950 in Buffalo, New York. From the early 1950's through 1978, Bengart & Memel received and dismantled polychlorinated biphenyl (PCB) transformers and capacitors, and during this process released PCBs into the soil. In the mid-1970's, soil samples from this site were found to contain greater than 50 ppm of PCB's. The New York State Department of Environmental Conservation issued a Consent Order for remediation, which required that the PCB concentration in the soil be reduced to less than 50 ppm.

Soil sampling and analysis indicated that seven locations on the property had excessive PCB concentrations. The PCBs were found at depths ranging from 6 to 24 inches at each location, and each location was found to be about 10 feet in diameter. As part of the remedial program, the contaminated soil was excavated and placed in 166 55-gallon steel drums.

Field Demonstration

The demonstration of the KPEG decontamination process took place during September and October 1986. Because of a consent order deadline, only 51 drums of soil were treated; the remaining drums were not processed but were shipped directly to a landfill.

KPEG treatment of the soil at Bengart & Memel involved six basic steps:

1) Setup of the vapor system and enclosure.
2) Addition of premixed liquid reagent to soil drums.
3) Attaching heaters, insulation, and vent lines to the drums.
4) Allowing the drum contents to heat and react.
5) Cooling and obtaining a sample for analysis.
6) Neutralization of the caustic soil.

The drums were filled with soil to within 2 to 6 inches from the top to allow space for the addition of the liquid reagents. An aluminum tube was driven through the dry soil to the bottom of the drum to allow air to escape during liquid addition; otherwise, an air pocket would form, preventing the downward movement of the liquid. A weighed quantity (typically 150-170 pounds) of a single-phase reagent mixture (2:2:4:9:5-PEG:TMH:DMSO:45% KOH: water) was added to each of the soil drums. The KOH reacted with aluminum; thus, when the reagent reached the bottom of the drum, foam exiting the tube would indicate that the air pocket had been expelled. The tube was then removed and a bimetal thermometer was inserted into the 3/4-inch bung on the drum cover.

The drums were heated from the bottom to ensure that all of the soil contaminants would react. For efficient heat transfer, it was essential that the liquid reagent in each drum remain in contact with the inside surface of each drum. The soil temperature was monitored 9 to 12 inches below the top of the drum, and the heater was positioned near the bottom of the drum. Typically, the soil would be maintained at temperatures ranging between 105° and 110°C for several hours.

The drums were vented to a central condenser system with a capacity of 16 drums (see Figure 10). During heating, vapors would exit from the drum through an insulated, flexible line connecting the 2-inch drum bung to the main header. Part of the header was ice-jacketed so that much of the water vapor was condensed and drained directly into a 55-gallon holding drum. The remaining vapor was drawn through an air-cooled radiator (used as a condenser), which also drained liquid to the 55-gallon holding drum. An ice-jacketed scrubber containing a dilute sodium hypochlorite solution (for odor control) trapped most of the remaining condensables. From the scrubber, the vapor passed through a 55-gallon drum, which was filled with a mixture of activated carbon and molecular sieve. A vacuum pump provided negative pressure for the vapor control system.

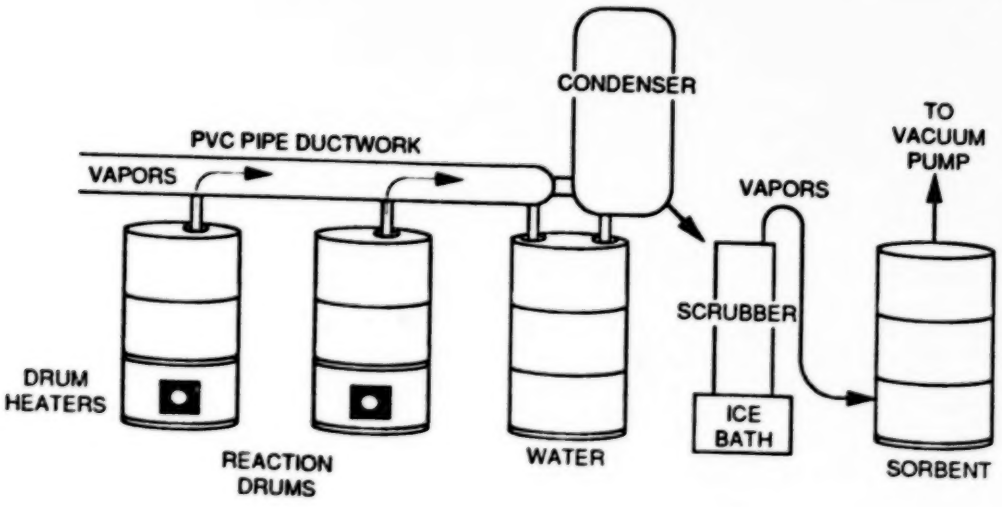

Figure 10. Central condenser system, KPEG drum treatment demonstration, Bengart & Memel, Buffalo, New York.

Treated soil samples from the drums were collected using a 30-inch auger attached to a hand drill. Four wells were made in each drum: one in the center, one close to the edge, and two spaced between the center and edge in different parts of the drum. Soil was collected by penetrating as deep as possible with the auger and bringing soil up from the bottom of the hole. In digging out a sample well, it was necessary to work around the large rocks in the drum--core samples were not obtainable. Debris (rocks, metal, wood, etc.) larger than 1 inch in diameter was not included in the sample. The soil from the cores was collected in a plastic bucket and mixed thoroughly with a trowel. Two jars were filled for each sample: one for GRC and one for the EPA. The soil remaining in the bucket was returned to the drum, and the auger, bucket, and trowel were cleaned before the next drum was sampled to avoid cross-contamination.

After the treated soil had been decontaminated, the soil had to be brought from highly alkaline to a neutral pH. Mixing was necessary to avoid "hot spots" of concentrated acid or caustic. To provide controlled mixing, the alkaline soil was carefully transferred to a drum partially filled with dilute acid. When the liquid became neutral or slightly basic, additional acid was added to compensate. Thus, as the acid and KOH reacted, there was sufficient mass to absorb the heat and prevent splattering. This procedure required frequent monitoring for pH control.

Results

Following KPEG treatment, the concentrations of PCBs were determined in selected drums. This group of drums, identified in Table 8 as Group 1, were

TABLE 8. CONCENTRATIONS OF PCBs FOUND IN UNTREATED AND TREATED SOILS BY EPA AND GRC ANALYSES, BENGART & MEMEL SITE (ppm)

	Group 1[a]		Group 2[b]		
Drum No.	Untreated	Untreated, 10/86	Untreated	Treated by EPA, 10/86	Treated by GRC, 2/87
1			138	71	19
4			84	60	20
5	119	43			
6	119	46			
8	62	45			
9			64	-	8
10	128	46			
11			128	90	30
20A			1300		<5
20B			1300		94
35	910	33			
36	91	46			
37			64	74	12
43			102		7
44			72	51	31
45			195	59	27
46	123	47			
53	64	15			
54A			78		10
54B			78		49
60	61	44			
62	65	30			
63			167		16
64			64		13
71	69	28			
72			119		12
81	74	38			
82	67	43			
83			78		18
85			78	56	16
86			84	74	22
87	62	39			
91	91	37			
92	138	49			
93	91	44			
101			74		11
102	69	18			
109	87	42			
111			64	51	22
112			110		33
113			67	78	17
114			102	57	27
119	64	9			
120			75	26	<5
121			62		24
122			106	65	22
131			78		17
135			106		28
137	62	23			
Misc A			NA[c]		33
Misc B			NA		17
Misc C			NA		9
Misc D			NA		6
Misc E			NA		15
Average	125	36	179	62	22
Average PCB Reduction		71.2%		64.4%	87.7%

[a] Analyzed by EPA.

[b] Analyzed by GRC.

[c] NA = Not Analyzed

sampled by the EPA soon after treatment. Those drums found to contain <50 ppm PCBs were neutralized without any further sampling or treatment.

A second group of drums (Group 2) was sampled and analyzed by GRC in February 1987. Group 2 included fresh samples from the drums that were found to contain over 50 ppm PCBs when EPA analyzed them in October 1986, samples from drums of treated soil that were never analyzed by the EPA, and one soil sample that was found to contain less than 50 ppm when the EPA analyzed it in October 1986. All of the drums in Group 2 were sampled in February 1987, after the reagent had been in contact with the soil for 5 months. The low-concentration sample was analyzed by GRC to check the potential reaction progress in drums with PCB concentrations below 50 ppm.

The analytical results of Group 1 and Group 2 data are summarized in Table 8

The reduced concentrations reported in February (compared with October results) indicate that the reaction continued during the holding period.

The only soil not to be reduced to below 50 ppm was from a drum of particularly high concentration (Drum 20 at 1300 ppm) that the site owner was not able to landfill. Drums 20 and 54 were processed in February 1987 during the neutralization procedures. The soil from these drums was split and transferred into four drums (20A and 20B, 54A and 54B) to ensure good reagent permeation and better heat transfer. In Drum 20B, the PCB level was reduced by 93 percent, to between 73 and 104 ppm. The levels in Drums 20A, 54A, and 54B were all reduced to less than 50 ppm.

In summary, the KPEG processing used at the Bengart & Memel site was successful in reducing PCB levels for 51 of 52 drums to below the 50-ppm control limit set for the site. For those 51 drums, the average PCB levels were reduced 75 percent, from 108 to 27 ppm. PCB levels for the sole remaining drum were reduced by 93 percent from 1300 to 78 ppm.

OMAHA, NEBRASKA, SITE

Contaminated Matrix Description

The purpose of this study was to demonstrate the effectiveness of the KPEG process for treating drummed, contaminated herbicide wastes containing dioxins. The study was conducted in 1987 at the site in Omaha, Nebraska. The contaminated waste contained 17,800 ppm 2,4-D, 2800 ppm 2,4,5-T, and 1.3 ppm 2,3,7,8-TCDD.

Field Demonstration

The KPEG reagent employed in this demonstration was prepared using polyethylene glycol and potassium hydroxide. The field-scale testing of the KPEG technology was performed on contaminated waste consisting of 20 gallons per batch. Only one batch of the waste was treated.

The treatment equipment consisted of a 55-gallon drum, a clamp-on heating band, and a stirring motor. The drum containing the KPEG reagent and the contaminated waste was heated to a temperature of about 70° to 85°C for 2 days, while being continually stirred. After 2 days, the heater was turned off and the reaction mixture was allowed to cool to the ambient temperature. The treated waste samples were then analyzed to determine the posttreatment concentration of the contaminated waste.

Results and Discussion

Table 9 represents the analytical results of samples collected prior to treatment and subsequent to the first and second treatments.

TABLE 9. DRUM-SCALE TREATMENT OF TCDD, 2,4-D AND 2,4,5-T

Contaminant	Conc. in untreated waste (ppm)	Concentration in treated waste: First treatment (ppm)	Concentration in treated waste: Second treatment (ppm)	Percent reduction of contaminants
TCDD (2,3,7,8)	1.3	0.086	ND[a]	100.00
2,4-D	17,800	9,000	334	98.12
2,4,5-T	2,800	1,700	55	98.04

[a] ND = None Detected. Detection limit unknown.

The KPEG treatment reduced the concentration of 2,3,7,8-TCDD in the waste to less than the detectable range. The concentrations of both 2,4-D and 2,4,5-T were reduced by 98 percent.

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